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British Steel in 1949

BRITISH steel production for 1949 has, as anticipated in our last issue, again exceeded the production target. At the beginning of that year the world's steel industry was still meeting with the abnormally high demand that had been encountered since the end of the war. War-time destruction was still being made good; arrears of work involving steel were still being tackled; and capital re-equipment, suspended during hostilities, was still proceeding. As the year progressed, however, the demand for steel arising from these and other causes abated, and in many countries the decline in demand was subsequently reflected in a reduction in output.

The steel industry in Britain was not immune from the change in demand evident elsewhere, though the change was less dramatic than in the world generally. Demand ceased to expand in all directions as it had done since the end of the war. The demand of some industries has become stationary, while that of others, shipbuilding for instance, is undergoing a decline. The problem of the industry now, therefore, is that of adapting production to these changing requirements. The volume of orders on the books of British steel companies reached its post-war peak in January, 1949. Since then there has been a fall, due, in the first instance, to the more rapid fulfilment of contracts, to the overtaking of demand by supply. From April, however, new orders, more particularly long-term than short-term orders, began to dwindle. The resulting decline in the course of the year in the total of steel orders on the books was about 20%.

For the greater part of 1949, however, the mass of orders awaiting execution was still heavy. Apart from the demand for home uses, steel was pressingly required by export trades called upon to make up for the decline of old export staples such as coal and cotton. About 30% of the entire demand was for steel for export directly or for indirect use in other export goods. This proportion is likely to be exceeded in 1950. A further indeterminate proportion was for the re-equipment of industries playing an important part in the export drive. Direct exports of steel were limited by Government policy.

The tasks set the steel industry, therefore, were, first, higher output, and, second, lower price. To the first the industry responded with the highest volume of production ever attained in its long history, its output exceeding the upper limit of 15·5 million tons which the Economic Survey of 1949 had estimated that the industry might attain in the *most favourable* circumstances. To the second it replied with prices which were among the lowest in the world. The actual steel production recorded is 15,552,900 tons, an all-time record for this country, and the comparison of prices shown in the accompanying table indicates that the British steel-using industries have an advantage over their foreign competitors.

UNITED KINGDOM AND FOREIGN HOME TRADE STEEL PRICES PER TON

Product	U.K.	U.S.A.	France	Belgium	Germany
	£ s. d.	£ s. d.	£ s. d.	£ s. d.	£ s. d.
Angles	19 3 6	31 4 0	25 0 11	24 3 4	20 0 11
Joists	20 3 6	31 4 0	21 12 4	—	19 2 9
Plates	21 14 6	31 12 0	29 3 0	25 18 11	22 10 6
Heavy Bars	21 13 6	34 0 0	25 0 11	24 4 9	19 19 2
Rerolled Bars	22 9 0	32 8 0	25 1 10	23 3 0	19 8 9
Hot-rolled Strip ..	23 4 0	34 16 0	26 1 5	25 16 8	23 17 9
Rails	19 2 6	27 8 0	24 2 9	25 8 0	21 4 2
Sheets	28 6 0	37 4 0	35 2 11	35 4 0	32 10 7

To-day the stocks of finished steel are the highest ever held in this country and supplies have grown plentiful and have caught up with, if not outdistanced, demand. Having fallen throughout 1947, stocks held by producers increased over 1948 by 150,000 tons and by September, 1949, this had risen to 175,000 tons. The growth of stocks in the hands of merchants, over the first half of the year, amounted to 60,000 tons, while consumers stocks rose 100,000 tons over the same period, after a rise of 350,000 tons in 1948. In the light of these figures the British Iron and Steel Federation is of the opinion that the period of shortage, as far as the home demand for steel is concerned, is over, the need of the immediate future is not so much an increase in the general supply as a flexible supply between the different finishing industries and firms. It is primarily on this ground that the industry, in the course of the year, sought the abandonment of all detailed control over distribution. As now worked the scheme still bears the marks of its war-time origins, when the Government was the main consumer, and the distribution of steel was controlled not so much by the issue of authorisations as by demands arising from programmes prepared by Government departments.

It is noteworthy, however, that four-and-a-half years after the end of the war, supply and demand in steel are once more practically in balance. That this is so is due to the industry's success in improving its capacity and in procuring the requisite supplies of raw materials, though the cost of these threatens to be one of the major difficulties of 1950 when production is expected to be increased a further 250,000 tons. Development schemes at Round Oak Works, Staffs., the Skinningrove Works, North East Coast and at Stewarts & Lloyds, Corby, will contribute substantially to this increase. The greatest increase in capacity represented by any development scheme will be that at Margam, this, however, will not be available until 1951.

As will be noted elsewhere in this issue the Economic Commission for Europe predicts a steel production surplus of 8 million tons in 1953, but the British Iron and Steel Federation itself believes it can market 16–18 million tons and its record of achievement entitles it to decide on what is practicable.

European Steel Trends

STEEL is a basic material on which virtually all phases of economic activity depend. Adequate, cheap supplies of steel are therefore essential for sound economic development, full employment, and a rising standard of living. This fact has been recognised by an impoverished Europe emerging from the second world war. National plans were made to overcome the post-war steel shortage which was holding up recovery, and to provide ultimately, through expansion of existing capacity, the steel necessary for further development. There was a danger, however, that such expansion plans, nationally conceived, would, taken together, prove mutually inconsistent, especially with respect to the prospects in export markets. Some light is thrown on the probable evolution of European steel production and consumption under present plans in a recently published report* prepared by the Steel Division of the Economic Commission for Europe which directs attention to some of these dangers.

One of the main conclusions reached after a study of the subject in relation to the world market is that current steel production plans in Europe are likely to result in a surplus of 8 million tons in 1953 which will not find a market. It is pointed out that if individual production of European countries (excluding the U.S.S.R.) are added together, total production in 1953 will be 70 million tons. Against this the conclusion is reached that the *practical* maximum consumption within Europe in 1953, provided it is a year of full employment, will be 58 million tons. An extensive survey of world producing and consuming areas further revealed that the maximum that Europe is likely to be able to export to oversea markets in 1953, is 4·1 million tons, which represents 80% of the estimated oversea import requirements of some 5 million tons. Thus the total demand for European steel in 1953 is not expected to exceed 62 million tons, leaving a surplus of about 8 million tons in crude steel.

In fact, as is pointed out, in order to achieve an efficient self-supporting European economy, which would enable a high level of steel consumption and enable Europe to recapture an 80% share of the world's steel markets, the prices of raw materials in Europe would have to be reduced and her production efficiency improved by suitable modernisation of plant and equipment. With the exception of manganese ore, Europe *as a whole* could be self-sufficient on an economic basis, so far as steel-making raw materials are concerned, and it therefore lies in her power to improve her efficiency and restore her competitive ability in world markets.

It is true that shortages persist in some countries for some kinds of steel, but these shortages are no longer due to a lack of production capacity. Payments difficulties arising either from a lack of money to invest in steel-consuming enterprises (as in Germany) or from a lack of suitable currencies with which to pay for requirements, still prevent potential demand from being met. Thus the European economy to-day presents a picture of unutilised capacity in the face of unsatisfied demand.

In tracing the development of the present position and the trends to which it is attributable, there is a growing tendency for individual countries to seek self-sufficiency in steel. It is recognised that, in the past, some steel-producing countries have used their position to exert political or economic pressure on those countries relying on them for supplies, and that this has inevitably tempted even small countries to build up national, frequently uneconomic, steel industries, protected by tariffs and other artificial barriers. Furthermore, the immediate post-war steel shortage tempted producers to take advantage of the situation, and to demand disproportionately high prices. This in turn was a further inducement to importers to create their own national steel industries.

No European country can effectively develop an independent and self-sufficient steel industry for the reason that each of them must remain dependent on some other country for its supplies of one or several of the raw materials necessary for the production of steel. On the other hand, the development of un-economic, protected, national steel industries can only have the effect of raising prices to a level which will reduce consumption and prevent a rise in the standard of living.

Discussing the consequences of 100% fulfilment of the present plans, the Steel Division Secretariat declares that in 1953 there would be produced a "cut-throat" competition amongst steel-producers which might well force out of business some efficient producers relying on a high proportion of export trade, while less efficient producers might continue to produce for national consumption at high prices, protected by tariff walls. It is pointed out that it is impossible to produce economically the whole range of basic steel products in a unit with much less than a 2½–3 million ton capacity per year—yet some countries with an annual consumption of half a million tons are planning their own steel production.

A considerable proportion of new steel production capacity is to be brought about by extending and modernising existing plants. It is estimated, however, that the present plans call for the building of entirely new steel works of a total effective capacity of 3–4 million tons. To construct a new steel plant with a capacity of 1 million tons a year would, in Europe, involve an investment of between 200 and 300 million dollars. It is obvious that money spent or to be spent on increasing production capacity which would create an unmarketable surplus of 8 million tons of steel, could be better invested in other more productive fields, and in particular in the development of steel-consuming industries.

Finally, suggested possible remedies for the problems facing the European iron and steel industry are proposed as follows :—

(*a*) Co-ordinated measures should be studied and applied in order to increase steel consumption when signs of under-consumption or recession appear ;

(*b*) Steel investment and production programmes should be co-ordinated in order to ensure an overall satisfactory level of crude steel production and an adequate supply in each of the main categories of rolled or drawn-steel products ;

(*c*) Co-operation among European countries should ensure adequate supplies of good-quality and cheap raw materials for steel-making ;

(*d*) Wide exchange of information on improvements in steel-producing techniques, on measures taken to increase productivity, etc., should be organised ;

(*e*) The evolution of the European and even of the world market situation from the point of view of steel production and consumption ought to be studied periodically so as to determine, in time, factors which might influence the decisions to be taken under (*a*), (*b*), (*c*) and (*d*) above.

* Report prepared by the Steel Division of the Economic Commission for Europe. Sales No. 1949, II. E.2. Published by the United Nations and available at H.M. Stationery Office.

The Hot-working of Nickel and High-nickel Alloys

The successful working of nickel and the high-nickel alloys Monel, "K" Monel and Inconel, probably more than most metals and alloys, depends upon correct heating, and in this article attention is directed to the means of heating, the time of heating and the temperatures at which the materials should be heated for various forging operations; important aspects of hot-working are then discussed to enable the development of techniques likely to ensure the most desirable properties.

SUCCESSFUL hot-working of nickel and the high-nickel alloys is largely dependent upon correct heating. This involves consideration not only of temperature and time required for heating but also of control of the furnace atmosphere.

Means of Heating

The most important difference between the heating of nickel and the high-nickel alloys and the heating of materials such as mild steel, chromium steel or some non-ferrous alloys is the greater susceptibility of the nickel-containing materials to attack by sulphur during heating. Metal surfaces which have been attacked by sulphur at high temperatures have a distinctly burned appearance and if the attack is severe, the material is weakened mechanically and may be rendered useless. Comparative specimens of Monel are shown in Fig. 7.

With nickel and the high-nickel alloys, exposure of the hot metal to sulphurous atmospheres or to other sources of sulphur must be scrupulously avoided. The most common source of sulphur is the fuel, and consequently care must be taken to avoid the use of fuels which have high sulphur content.

Electricity.—Although electric resistance furnaces are ideal for annealing operations, the temperatures to be attained for hot-working of the high-nickel alloys preclude the use of the standard type of furnace with 80/20 nickel-chromium metallic resistors. Even furnaces with other types of element are unlikely to be competitive in cost with gas or oil heating.

Heating by induction methods, as applied in the forging of steel, while equally applicable to high-nickel alloys, is at present only likely to be economic on fairly long runs of similar size blanks or billets.

Gas.—Gas is the most desirable fuel for heating Monel, nickel and Inconel, and should always be used if available. Gas fuel requires only a small combustion space and permits easy automatic regulation of temperature and of atmosphere within the heating compartment. The convenience, simplicity and ease of control in modern furnaces equipped with mixers which automatically proportion the gas/air mixture over the full range of heating give important advantages to gas in terms of good heating.

Town gas which is low in sulphur is a satisfactory fuel for Monel, nickel and Inconel. The maximum permissible amount of sulphur depends on whether furnace conditions are reducing or oxidizing. In oxidizing conditions 25 grains per 100 cu. ft. of gas is permissible but under reducing conditions the figure should be 5 grains per 100 cu. ft. or less. Under any conditions the lower figure should be aimed at if at all possible. Various methods are available for purifying town's gas from sulphur, such as the Gas, Light and Coke Company's catalytic process, the Maxted catalytic process, or treatment with activated carbon. Gas is used especially in small furnaces for heating small forgings, ornamental work, bar ends for hot-heading rivets and bolts, and for other light work. Its good qualities justify its higher cost per B.Th.U.

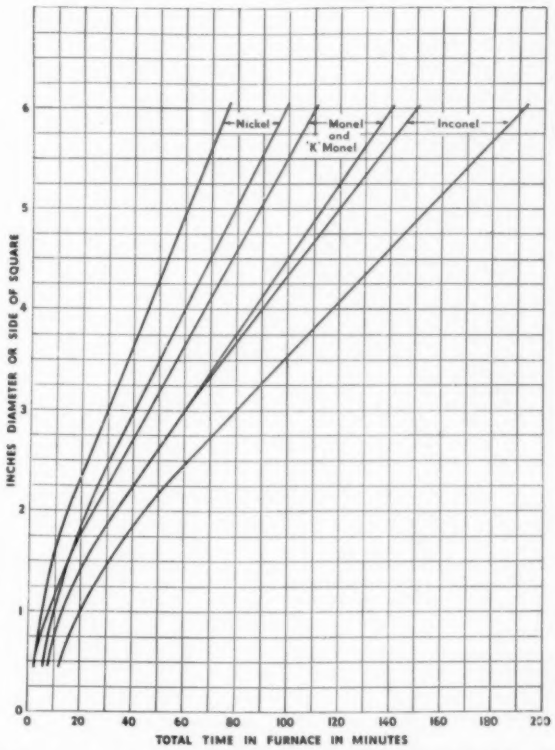

Fig. 1.—Approximate time required for heating Monel, nickel and Inconel bars of different sectional dimensions.

Butane and propane are also very desirable fuels. While their cost on a B.Th.U. basis is higher than that of oil, the ultimate cost per pound of finished forgings may easily be lower. Both gases are obtainable in cylinders equipped with pressure regulators to control the flow of gas and in this form are particularly useful for occasional work in heating Monel, nickel and Inconel for forging, hot-working and riveting.

Oil.—Fuel oil is most generally used for heating the high-nickel alloys in the smiths' shop. It should be specified to have a sulphur content of not over 0·5%,

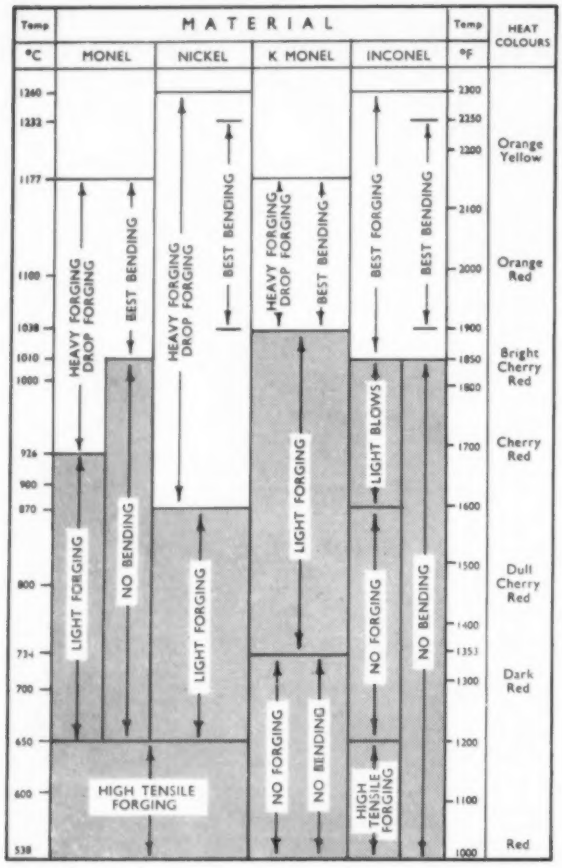

Fig. 2.—Approximate temperature ranges for hot-forging and bending nickel and various nickel alloys.

though the heavier oils purchased under such a specification seldom exceed 0·2% sulphur and the lighter distillates are almost sulphur-free. The actual permissible figure depends largely upon furnace conditions, for while the slightest trace of sulphur is most detrimental in a furnace atmosphere that is reducing, in oxidising conditions sulphur in small quantities has little or no detrimental effect on the metal being heated.

Any well-designed burner that will mix thoroughly the oil and air supply and that can be closely regulated can be used. Burners which utilise low-pressure air, supplied through the burner, are more satisfactory than the high-pressure air or steam-injector types and work well with temperature-control equipment.

Coal and Coke.—Coal and coke are generally unsatisfactory for heating nickel and high-nickel alloys. Although on a B.Th.U. basis coal is in most localities the cheapest source of heat, it is almost certain to be the most expensive when compared with oil, gas or even electricity on the basis of total heating costs, which include rejections, additional metal required to provide for scaling losses and higher finishing costs.

Solid fuels are unsatisfactory because of the difficulty of obtaining proper heating conditions, inflexibility in heat control and the presence of excessive amounts of sulphur. While it is not impossible to heat the high-nickel materials with the solid fuels such as "blacksmith" coal or coke from coal treated to reduce sulphur, it is very difficult to obtain satisfactory results and the practice is not recommended.

Avoiding Sources of Sulphur

Mention has already been made of the need to avoid exposure of the high-nickel alloys to sulphurous atmospheres at high temperature and of the influence of various types of fuel in this respect. But in addition to the fuel there are other potential sources of sulphur which must be considered.

Scale, slag and cinder on the hearth of the furnace may contain sulphur. To prevent contact with these, the work should be supported above the hearth on rails or supports. The possibility of the furnace brickwork containing sulphur from previous operation on high-sulphur fuels should also not be overlooked. Sulphur may also be introduced in the form of sulphate salts and sludge remaining on the work from a prior pickling operation or in oil and lubricants used for machining or other fabricating operations. All such contamination should be completely removed before heating. It is also advisable to remove all marking paint, which frequently contains metallic sulphide pigments that can cause embrittlement of the painted areas.

Heating Temperatures

Approximate temperature ranges for hammer-forging, die-forging and bending are shown in Fig. 2. It will be noted that Monel, "K" Monel, nickel and Inconel show important differences as to proper hot-working temperatures, particularly in the upper safe heating limits and tendencies towards reduced hot ductility.

While the high-nickel materials are being heated the furnace temperature should be held about 30° C. higher than that at which the work is to be "pulled" and in no case should it be pulled at temperatures below 1,100° C. The practice of working the furnaces at excessive

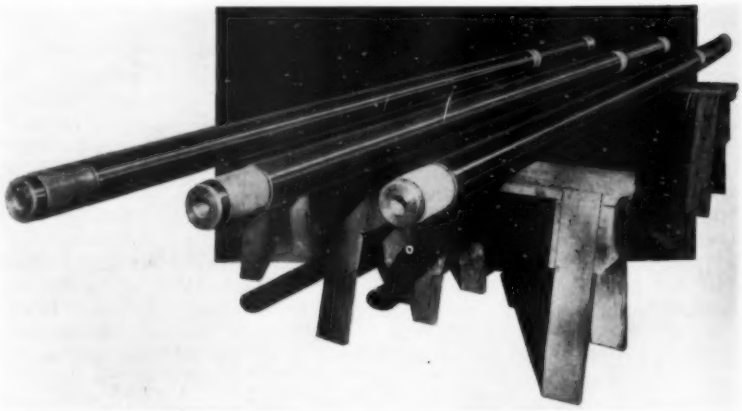

Fig. 3.—The high strength of these forged Monel gate stems (6 in. dia × 18 ft. 4 in. long) was obtained by hammering below the useful forging temperature range.

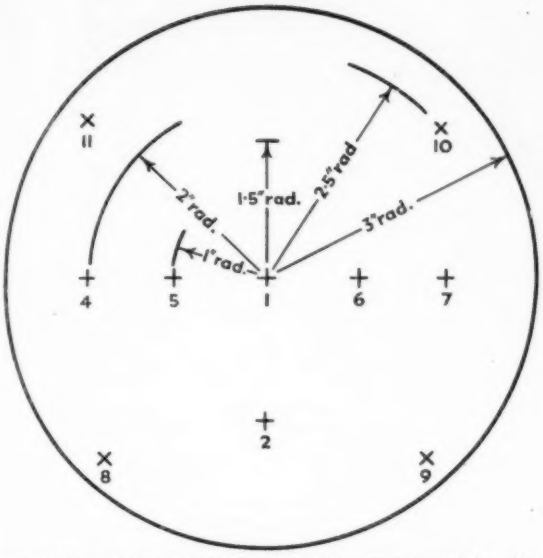

Fig. 4.—Location of the test pieces in sample of 6 in. dia. gate shaft shown in Fig. 3. Results of these tests are given in Table II.

temperatures in order to reduce heating time is damaging to nickel and high-nickel alloys. Results following the use of correct and incorrect heating temperatures are illustrated in Fig. 7.

Though many shops heat nickel and nickel alloys successfully without the aid of pyrometers or automatic temperature control, it is wise to provide these facilities. In their absence, however, preliminary trials should be made on scrap pieces to assist the judging by colour of proper temperatures for hot-working. A useful bend test is described in a succeeding page, while Table X, which will give data on suitable thermocouples, will be published in a succeeding issue.

Fig. 6.—Cold-bend tests on Monel rivet stock. The 180° bend was obtained on a piece heated in a sulphur-free atmosphere and the surface shows no sign of defects. The other piece which was heated in a pea-coal-fired furnace shows pronounced traces of sulphur embrittlement. These are shown clearly in the enlarged section above.

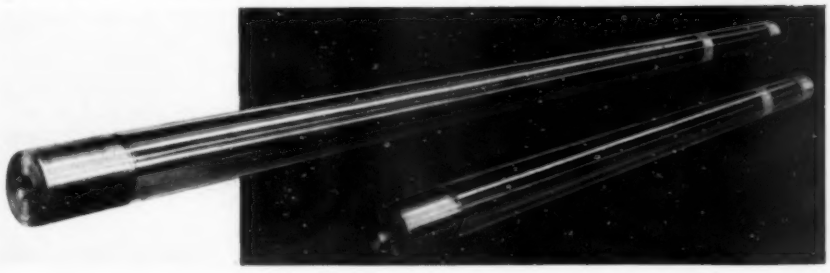

Fig. 5.—These hammer-forged, heat-treated and machined "K" Monel stems supplied for the Tygart River Dam have the mechanical properties shown in Table III. The shorter stem is 6 in. dia. × 12 ft. 9 in. long; the longer stem is 7 in. dia. × 18 ft. 5¼ in. long.

Time of Heating

Monel, nickel and Inconel should be heated for a time just sufficient to ensure that the desired temperature is reached uniformly throughout the piece. It is bad practice to expose these materials to hot combustion gases for long periods after the pieces have reached a uniform hot-working temperature.

It is not possible to give rules to govern the time of heating because furnaces and furnace operation vary so widely. Rough approximations of the total time desirable between charging and drawing the work are shown in Fig. 1. It should be noted that short slugs receiving heat from all sides require from 30–50% less time, while flat slabs and plates require 50–100% additional time.

When the furnace is to be charged with several pieces of metal the work should be so arranged that each piece will be withdrawn immediately it is heated uniformly to the required temperature to avoid holding metal in the furnace after it is ready for forging.

TABLE I.—RANGE OF TENSILE PROPERTIES FOR FORGINGS

	Maximum Stress Tons/sq.in.	0·5% Proof Stress Tons/sq.in.	Elongation % on 2 in.	Reduction of Area %	Hardness D.P.N.
Monel	36–49	18–38	40–20	70–50	140–230
Nickel	29–47	9–36	40–25	70–50	97–215
Inconel	38–54	16–40	45–20	70–50	145–230
"K" Monel:					
as forged	46–54	18–40	40–25	—	150–240
heat-treated ..	60–74	45–56	30–20	—	260–310

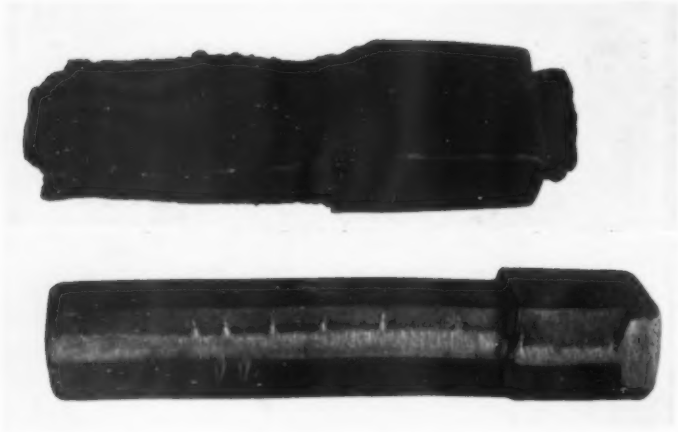

Fig. 7.—The Monel bar upper was heated to a temperature above the safe limit for forging. The lower one was heated to about 1,180° C. (2,150° F.) which is within the safe range.

TABLE II.—PROPERTIES OF MONEL GATE STEMS (Figs. 3 and 4)

Test Piece No.	Yield Point Tons/sq. in.	Maximum Stress Tons/sq. in.	Elongation % on 2 in.	Reduction of Area %
1	33	44	34·5	60·4
5	33	45	33·6	60·4
6	33	45	33·6	60·4
2	35	46	32·8	60·4
3	35	45	34·5	57·9
4	35	45	33·6	59·8
7	33	45	35·5	63·6
9	34	45	34·5	63·1
8	34	46	30·1	65·0
10	34	45	36·0	63·1
11	34	45	34·0	61·8

TABLE III.—TENSILE PROPERTIES OF FORGED HEAT-TREATED "K" MONEL GATE STEMS (See Fig. 5)

Limit of Proportionality ..	Tons/sq. in.	40
0·2% Proof Stress	Tons/sq. in.	49
Maximum Stress	Tons/sq. in.	68
Elongation	% on 2 in.	24·7
Reduction of Area	%	41·6
Hardness	D.P.N. ..	300

In heating for small drop forgings weighing 5 lb. or less, or for upsetting bar ends for rivets and bolts, a small number of pieces should be charged first and whenever one is withdrawn to be forged a cold piece should be put in the furnace. The number of pieces to be charged at the start is determined by the time required to bring a freshly charged piece to the forging temperature and by the number of pieces to be forged in a unit of time. For example, in hot-heading ¾ in. dia. nickel rivets at a rate of 3 per min., the bar end may require 10 min. in the furnace to reach the proper forging temperature; hence the rate of forging will be thirty times the rate of heating. If 30 pieces be charged all at once, the heading machine can be started working steadily after 10 min., but the last piece will have been ready for forging 10 min. before it is pulled. If 60 pieces be charged, the last piece pulled will have been at forging temperature for 20 min.

The nickel alloys cannot be heated successfully in this manner. The right method for the above conditions is to charge at the rate of three rods per min. After 10 min. heating, the first rods will be ready and as one is pulled a cold piece can be charged. The heading machine can then be put into steady operation.

Larger work which may require as long as 20–30 min. to reach the required temperature and perhaps a minute to forge should be charged in the manner outlined.

In some shops furnace capacity is inadequate to keep the hammer or heating machine in steady operation. This is especially noticeable with nickel, for which the flame temperature must be lower than for steel. This means that a longer time is required to bring the metal to forging temperature and the production per machine will necessarily be less than when forging steel.

Furnace Atmospheres and their Control

The furnace atmosphere in the vicinity of the work, which in practice means the gases of combustion sweeping over the metal surface, requires careful control. The tendency to absorb sulphur from furnace gases is more pronounced in a reducing atmosphere than in a slightly oxidizing atmosphere. If the fuel contains sulphur it is necessary to work the furnace with a slightly oxidizing atmosphere and to tolerate a certain amount of scaling rather than to operate with a reducing atmosphere.

In heating for hot-working, however, it is desirable to avoid scaling. Provided that sulphur-free fuel is available, the atmosphere should be adjusted so as to contain 2% or more of carbon monoxide. In general, slight excess of fuel over air is all that is required and the closer the atmosphere is to the neutral condition, the easier it is to maintain the required uniform temperature. The atmosphere should not be permitted to alternate from reducing to oxidizing. The slightly reducing condition desired is obtained by cutting down the air supply until there is a tendency to smoke, indicating an excess of fuel and a reducing atmosphere. The air supply should then be increased slightly to give a hazy atmosphere or a soft flame.

The actual condition of the furnace atmosphere can be determined by analysing samples of gas taken at various points about the metallic surface.

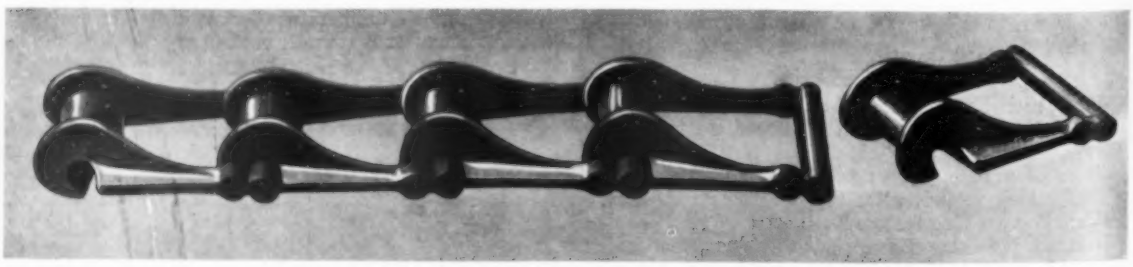

Fig. 8.—Drop-forged Monel links for conveyor chains used in pickling plant.

The stack dampers should be closed wholly or partially in order to force the gases out under the furnace door, where the excess of unburned fuel will burn freely. The slight pressure thus set up in the furnace prevents the entrance of air under the door or through the charging slot.

Care should be taken to bring the furnace up to the upper limit of the hot-working range before introducing the parts which are to be heated. Once the furnace attains the full temperature, it should be adjusted to maintain the temperature with complete combustion close to the burners. It is important that combustion reactions be complete before the resulting gases come into contact with the work, otherwise it may suffer damage through embrittlement.

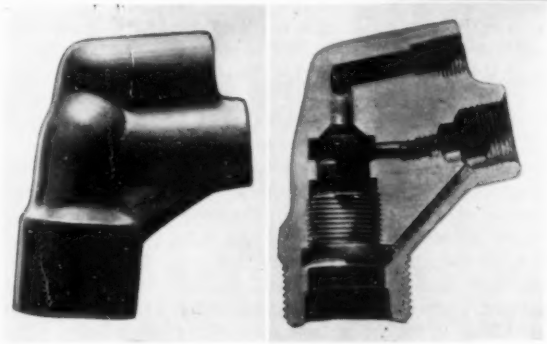

Fig. 9.—Hot-forged Monel head for oxy-acetylene cutting torch.

This consideration is particularly important when oil-fired furnaces are used, for oil fuel requires more combustion space than most gas fuels.

In the slot-type oil-fired furnace with burners in the back or end walls, the combustion zone is sometimes insufficient to permit complete combustion before the gases reach the work. Indeed, it sometimes happens that a large proportion of the oil is gasified by impingement on the work in the heating zone. High-nickel alloys thus placed directly in the unburned fuel are subject to damage and must therefore be shielded by steel plates.

Hot-working—Forging

If correct heating practice has been followed, Monel, "K" Monel, nickel and Inconel can be forged, in dies or by hand, into products of the highest quality and into almost any shape that can be forged in steel. Die forgings such as conveyor chain links and hooks are made regularly and hammer forgings of many types are produced in a great variety of forms. These include large-diameter bars for shafting and hollow-bored tubing, seamless forged rings, pickling hooks, etc. In addition, pipe fittings, valves and the like, can be produced as hydraulic or mechanical press forgings.

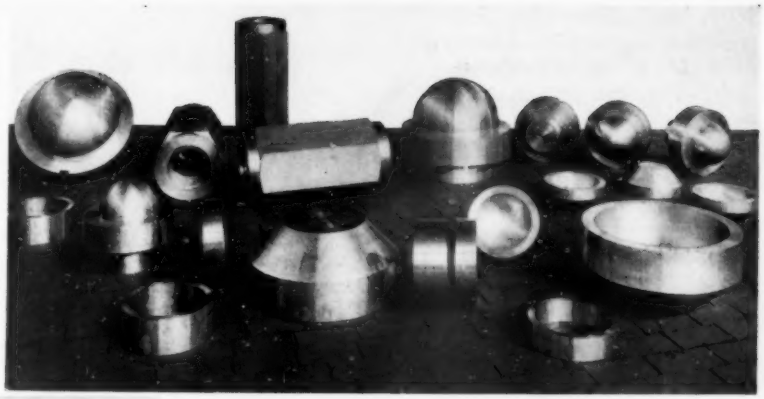

Fig. 10.—A group of machined Monel hammer forgings.

Forgings of Monel, nickel and Inconel are stronger than those of bronze or of carbon steel that has not been subsequently heat-treated. Corrosion-resistant forgings for applications where unusually high mechanical properties are required can be made from "K" Monel, which can be hardened by thermal treatment. Forging characteristics of this material do not differ materially from those of ordinary Monel, but certain special points are discussed later.

Checking Heating Conditions and Forging Temperatures

Before charging work to the furnace, it is advisable to check the correctness of the heating conditions. A test can readily be made by heating a bar of convenient size, say $\frac{1}{2}$ in. diameter round rod or $\frac{1}{2}$ in. $\times$ 1 in. flat bar, to the required temperature and holding it in the furnace for 10–15 mins. The piece should then be either water quenched or air-cooled, and bent through 180° flat on itself. If the conditions of heating have been correct there will be no sign of cracking. The extent of any damage that may have occurred is judged by the depth of the cracks. If the operator has no experience with the high-nickel materials, and there are no facilities for automatic control of temperature, it is advisable to practise drawing out bars under the hammer, taking care to preserve square edges and then to make bend tests to gain familiarity with the temperature range in which the metals may be forged safely. Bend tests are best made on bars approximately 1 $\times$ $\frac{1}{2}$ $\times$ 6–8 in. long. A good practice is to charge several of these test pieces into the furnace at a time and pull single bars at various temperatures as they are being heated up. Each bar should be bent immediately through a

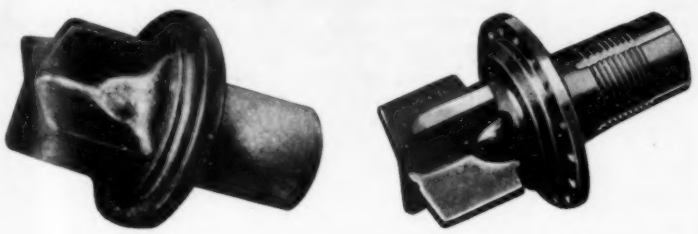

Fig. 11.—Drop-forged Monel disc for high-pressure steam pop safety valve. At left is shown the trimmed die forging and on the right the finished part.

180° bend. This may be started in a "vee" block and finished by flattening down under the hammer, or the bending may be done by hand. It must be done as rapidly as possible to avoid an excessive drop in temperature. When the forging temperature is correct there will be no sign of cracking.

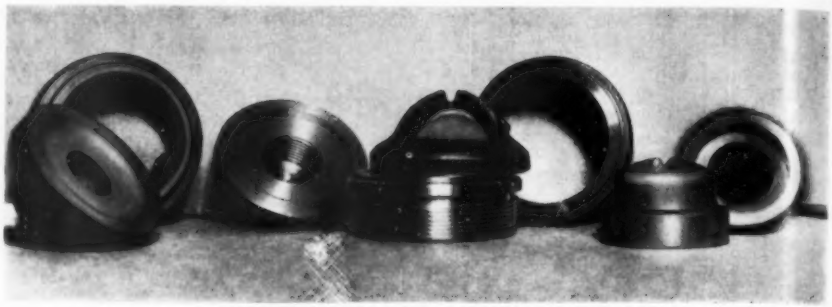

Fig. 13.—Drop-forged steam valve trim. The two pieces at the right end are stellite-faced, ordinary Monel. The other six pieces are solid "K" Monel. Hardnesses up to 320 D.P.N. are obtainable in such forgings of "K" Monel.

Fig. 12.—Hammer-forged Monel hooks used for moving large pickling crates and other heavy loads.

Forging, Pressing and Bending Operations

If the work has been heated properly and the working temperature ranges shown in Fig. 2 have been observed, there will be little difficulty in forging, pressing or bending. Nickel and high-nickel alloys are considerably stronger and stiffer than plain-carbon steel at forging temperatures and thus a machine worked at full load to produce a given steel component may be incapable of producing the same forging in these materials. It may, therefore, be necessary to use heavier hammers and larger machines than would be required for handling mild steel. This applies particularly to single-blow operations, as in heading rivets and bolts and in pressing.

"K" Monel needs special mention for, while general hot-working procedure and temperature ranges are similar to those already described for ordinary Monel, it must be remembered that "K" Monel is a heat-treatable alloy. Consequently, if "K" Monel is allowed to cool in air from the forging temperature, it may become too hard for cold-forming or working in later operations such as machining.

The following points should therefore be noted :—

1. If it becomes necessary to re-heat the metal in order to complete the work, it should be re-charged into the furnace with a minimum of delay.

2. When hot-working has been completed, or when it is found necessary to allow the metal to cool before further hot-work, the metal should be quenched from 790°/800° C. (1,450°/1,470° F.) or above. It should not be allowed to cool in air. The preferred quenching medium is water, to which about 2% by volume of alcohol has been added. Unless it is quenched, the piece will self-heat-treat to some extent on cooling, setting up stresses that may result in cracks or in surface tears either during cooling or subsequent heating.

3. "K" Monel which has been allowed to air-cool from around 700° C. (1,290° F.), instead of having been quenched as described above, may be too hard for practical cold-forming or working later.

4. It is not advisable to attempt to increase the strength and hardness of "K" Monel forgings by subjecting them to semi-cold forging. Such operations are likely to result in damage to the material, due either to forging in the range of low ductility or to the possible effects of self-heat-treatment. Moreover, there is no practical advantage to be gained by such forging after heat-treatment, because the properties of a hot-finished and a semi-cold-finished forging will be found to be essentially the same.

Dies and Lubrication

Die blocks for drop forgings or pressings may be made from either straight carbon or alloy steels. The choice between plain-carbon steel and one of the several excellent alloy die steels available will depend largely on the shape and size of the piece, the quantity to be produced, and the material to be forged. Dies of 0·80% carbon steel, hardened and tempered to 450 D.P.N., can be expected to give a good life on the smaller forgings in nickel and Monel. There is no point in heat-treating carbon steel dies above 450 D.P.N. as the heat of the hot metal being forged will always reduce the hardness and may crack the die. Alloy-steel dies meet the needs of modern production better than do carbon-steel dies, and their use is almost a necessity for the larger or more intricate shapes in Monel or nickel, and for all forgings in Inconel except small, simple shapes.

Trimmer dies should be made of high-speed steel, with the cutting edge ground to a considerable rake. They should be set up closely enough to cut tissue paper. The trimming may be done either hot or cold.

There is a slightly greater tendency toward sticking in the die with the high-nickel materials than with steel, especially with difficult shapes. A swab with a mixture of heavy grease and graphite ensures proper clearing of the work from the die. Sawdust thrown into the die is also useful in preventing sticking. It is recommended that lubricants be sulphur-free.

Attention is drawn to the advisability of having dies pre-heated before forging these materials. Cold dies will

chill the surfaces of the forging excessively and in the case of intricate shapes may cause rejection of the first few pieces.

Strength of Forgings

Tensile properties of nickel and the high-nickel alloys are given in Table I. The maximum stress and proof-stress values will approach the higher figures when forging is carried out in the lower part of the temperature ranges recommended (see Fig. 1). The strength of hammer forgings may be materially improved by judicious hammering after the material has cooled to 650° C. (1,200° F.). This is illustrated by the values given in Table II for some large, high-strength Monel sluice gate stems. The locations of the individual test pieces in the forged bar are shown in Fig. 4.

The unusually good properties available in heat-treated forgings of " K " Monel are shown in Table III, which gives values obtained on test pieces taken from the hammer-forged " K " Monel gate stems shown in Fig. 5. Forgings of " K " Monel are heat-treated by holding at 583°–593° C. (1,075°–1,100° F.) for 8–16 hours after the entire mass of metal has attained a uniform temperature.

Chemico-Mechanical Grinding of Hard Metal Tips*

Following a discussion of the importance of surface grinding the supporting surfaces of hard metal tool tips before brazing, an account is given of a combined chemical and mechanical method of carrying out the grinding operation.

THE grinding of the underside of hard metal tips by silicon-carbide grinding wheels is not widely used owing to the low efficiency of the method. Because of the non-magnetic nature of the hard metal, the bits have to be fixed to the machine by clamping, the use of melted sulphur or resin being inconvenient. For these reasons it is often preferable to use unground tips for brazing.

Brazing is improved merely by removing the surface oxide layer and for this purpose tips are sometimes placed in a container into which they fit accurately, or into a recessed wooden bar. As the attachment to the wooden bar is not perfectly rigid, however, a curved surface is quite likely to be produced on the tip.

Under practical conditions the thickness of the layer of brazing metal (copper) should not exceed 0·1 mm. (1 mm. is approximately 0·040 in.). Figs. 1–3 show that, with tips 7 mm. thick, surface inequalities may reach 0·1 mm. In such a case, and with a curved underside to the bit, the layer of brazing metal under the front part of the tip would become 0·2 mm. (Fig. 2). If such a tip lies edgewise on the supporting surface, the thickness of the layer may be increased to 0·3 mm. With thin plate tips, curving of the underside is considerable and, furthermore, the layer of brazing metal is thicker; it may be as much as 0·5 mm.

From Fig. 2 it will be seen that, with a thick layer of copper under the front of the tip, the latter may not be adequately supported and, under pressure of the chips in cutting, cracking of the tip or displacement from the shank may occur. With multi-point tools it is desirable that hard metal tips should have not only even surfaces, but also strictly parallel ones, otherwise there is a further risk of displacement in heat treatment. The following methods of overcoming these difficulties have been tried:—(*a*) The hard metal tip is fastened to the tool shank by wire—this was found to be inconvenient for further processing; (*b*) the tool is coated with clay in a special device—this increases the cost; (*c*) an oxy-acetylene flame is used for brazing each tip separately—trouble is experienced due to increased air cooling of the sintered parts brazed first.

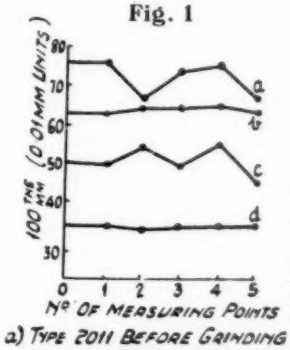
100 THS MM (0·01 MM UNITS)
Nº OF MEASURING POINTS
a) TYPE 2011 BEFORE GRINDING
b) TYPE 2011 AFTER GRINDING
c) TYPE 44 BEFORE GRINDING
d) TYPE 44 AFTER GRINDING

Fig. 1

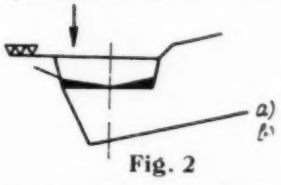

Fig. 2

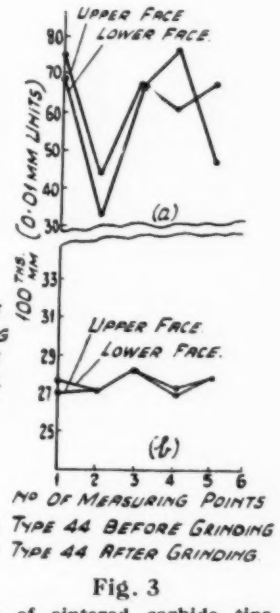

Fig. 3

Fig. 1.—Surface roughness of sintered carbide tips. Fig. 2.—Effect of insufficient support of sintered carbide tips. Fig. 3.—Surface roughness of upper and lower faces of carbide tips.

The difficulties mentioned may be overcome by a combined chemical and mechanical method. When a hard metal with a cobalt base is placed in a solution of copper sulphate, cobalt displaces copper, forming cobalt sulphate, and a thin layer of copper is formed on the hard metal. This protects the alloy from further action, but if the film is removed the reaction recommences. For the rapid removal of the copper film, the State Optical Institute (U.S.S.R.) uses a glass grinding machine (Fig. 4). A copper disc of 120–150 mm. diameter and 10–20 mm. thickness is heated by an electric plate to 60° C., the melting point of a mixture of three parts by weight of wax to one part of rosin (colophony). This mixture is spread evenly over the disc to a thickness

* Kolker, B. M., *Stanki i Instrument*, **20**, 1949, 5, 17–19.

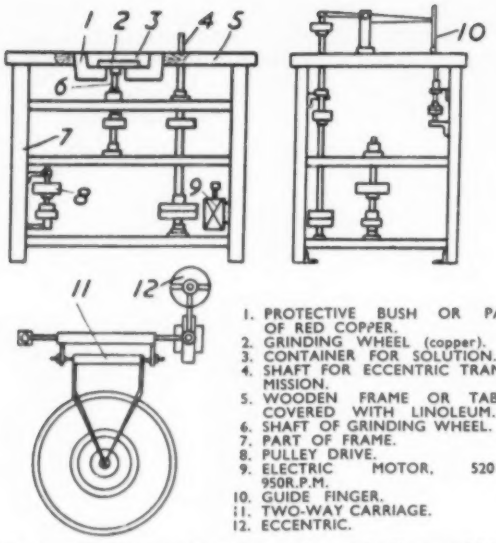
1. PROTECTIVE BUSH OR PAN OF RED COPPER.
2. GRINDING WHEEL (copper).
3. CONTAINER FOR SOLUTION.
4. SHAFT FOR ECCENTRIC TRANSMISSION.
5. WOODEN FRAME OR TABLE COVERED WITH LINOLEUM.
6. SHAFT OF GRINDING WHEEL.
7. PART OF FRAME.
8. PULLEY DRIVE.
9. ELECTRIC MOTOR, 520 v., 950R.P.M.
10. GUIDE FINGER.
11. TWO-WAY CARRIAGE.
12. ECCENTRIC.

Fig. 4.—Special machine for surface grinding carbide tips.

of 0·5 mm. and the tips are placed on this under slight pressure. After packing the tips on the copper disc, the whole is left for the wax to set. Copper vessel 3 contains 100–200 ml. of 25% copper sulphate solution together with 50–100 g. of "five-minute" emery. The copper disc with the hard metal tips is so disposed that the tips are located between the grinding mechanism and the disc. Part 10 (finger) of the movable carriage is inserted in the opening in the disc. To this finger is attached a load of about 0·3–0·4 of the maximum possible load required for grinding, as determined by Fig. 5. The full load is not applied until the machine has been started. When container 3 is rotated, the solution therein is centrifuged to the walls of the vessel and returned to the centre by means of baffles radially disposed between the disc and the wall of the vessel, and extending as low as possible without touching the bottom.

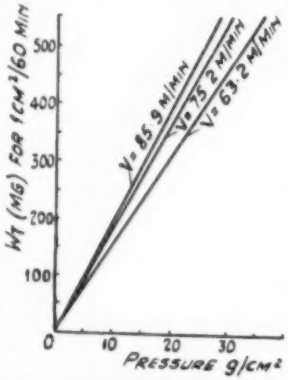

Fig. 5.—Performance of grinding machine for different peripheral speeds.

After completion of grinding, waste material or the like adhering to the surface must be removed, since cleanliness is essential to hard soldering. The guide plate or baffle is taken out and the grinding parts washed for a minute or two with clean water and emery powder. To secure uniform wear of the grinding part, carriage 11 is connected with eccentric gear 12, which transmits to both disc and tips a slight to-and-fro motion, at a rate of some 60–80 strokes per minute, this being regulated according to the nature of the abrasion; if concave, the rate is increased and *vice-versa*.

A rotary movement of the upper disc is obtained by a frictional engagement with the grinding wheel, so that the hard metal, during grinding, has two motions—i.e., rotary and linear. Abrasion is thus both slow and uniform, and the grinding wheel retains its shape and efficiency for a long time, redressing in the lathe being required after about 200 hours of operation. It is possible to grind 80–100 tips simultaneously, the time for one batch being 30 minutes, after which the disc is removed from the machine and heated for removal of plates, these being cleaned in kerosene.

Up to a certain point, the output of the machine increases in direct proportion to the load on the disc and the speed of rotation. With V=86 m./min. the centrifugal force is such that the emery is only retained on the grinding wheel with difficulty. Removal of the film begins to slow down, and the machine gets clogged or soiled with sprayed liquid. Increasing the load beyond 40 g./sq. cm. is also undesirable owing to a certain grinding down of the emery which reduces its power to remove the film. Generally a maximum load on the disc of 20 kg. is sufficient.

With this method it is possible to obtain hard metal tips with parallel even faces which can be tightly pressed together in an assembly of multi-cutter tools, no supplementary measures being needed to fix the tips for hard soldering in a furnace. The method is mechanically sound and reliable. The use of small-diameter multi-cutter tools has its limitations, since some thicknesses and lengths of tip required cannot be achieved easily. For instance, with broaches or reamers of 6–8 mm. diameter, tips of 0·5–0·8 mm. thickness are required. The method described makes it possible to obtain tips of the desired minimum thickness and with a high degree of accuracy (down to 0·01 mm.).

"Research and Production" Exhibition

An exhibition entitled "Research and Production" is to be held by the British Welding Research Association at their London Headquarters, 29, Park Crescent, W.1, from Tuesday, February 7th, 1950, to Saturday, February 11th inclusive, between 10 a.m. and 5-30 p.m.

Every aspect of welding will be covered by the exhibition which is to show how research has helped welding, and how welding can increase productivity.

Stands have been allotted for the following:—Welding stainless steel; welding magnesium and its alloys; welding aluminium and its alloys; welding steels; non-destructive examination of welds; repairs by welding; resistance welding; resistance welding machine instrumentation; welded pipes and pressure vessels; and welded structures.

Demonstrations will be given of the non-destructive testing of welds and resistance welding machine instrumentation. Films will be shown daily and a number of lectures are being arranged.

An Information Department will be in operation and with the aid of engineers of the Liaison and Development Department it is hoped to answer queries regarding the many applications of welding.

Telegraphic Address

The Rapid Magnetting Machine Co., Ltd., Lombard Street, Birmingham, 12, manufacturers of permanent and electromagnetic separating and lifting equipment, inform us that their telegraphic address will in future be Magnetism Birmingham, 12.

Creep and Fatigue Tests on Commercially Extruded Lead and Lead Alloy Pipes*

By J. McKeown,† D.Sc., M.I.Mech.E., F.I.M. and
L. M. T. Hopkin,§ B.Sc., A.R.S.M., A.I.M.

(Communication from the British Non-Ferrous Metals Research Association.)

Examination of service failures of lead and its alloys indicates the importance of a knowledge of the creep- and fatigue-resistance of these materials. The work recorded in this report was intended as a preliminary investigation to determine the degree of reproducibility to be expected from materials produced by extrusion on commercial presses, as it is well known that the creep properties, in particular, are very sensitive to variations in manufacturing conditions. It is concluded that work of a basic nature on the effect of alloying additions on the creep and fatigue properties cannot be carried out on extrusions made on commercial presses, where the many variables are not under sufficient control. The first part of this report is presented here, the second and concluding part will be published in our next issue.

FOR a number of years before the last war the British Non-Ferrous Metals Research Association had been carrying out investigations on the mechanical properties of lead and lead alloys extruded in the form of water pipe, rod, and cable sheathing. In most cases the basis lead was Broken Hill of ordinary purity and in every case the materials were extruded on commercial presses.

Most of these investigations were concerned with the resistance to creep and to fatigue of the basis lead and with the effect of alloying additions on these properties. Some of the work carried out on the basis lead has already been described.[1]

At the commencement of the war the experimental work had led to the following conclusions :—

In any one lead alloy grain size is the factor that exercises the greatest influence on creep resistance at moderately low stresses ; fine grain size gives rise to rapid creep and a coarse grain size to slow creep. The grain size is, in general, a function of the temperature of extrusion, being greater the higher the extrusion temperature. For a given extrusion the temperature an alloy has a finer grain size than the basic pure lead. For specimens of the same grain size, alloys have a much higher resistance to creep than pure lead. Cold working, such as occurs during the coiling and uncoiling of pipe and cable sheathing, can influence the shape of the creep curve obtained for lead.

In general the higher the alloying addition, the greater the endurance limit in fatigue of the alloy relative to pure lead.

In both lead and lead alloys the resistance to fatigue is greatly increased by the presence of a protective coating.

The effect of grain size on the resistance of a lead alloy to reversed stresses is not clearly established but appears to be linked with the rate of reversal.

The understanding of the creep of lead alloys resulting from this early work found application in the development of delay action fuse mechanisms.[2] The present paper describes more recent work on the creep and fatigue resistance of alloys in industrial use for pipes and cable sheathing.

An examination has been made of materials extruded in the form of pipe on two main types of extrusion press, the standard inverted extrusion pipe press and the newer form of continuous press which is finding increasing application in the production of cable sheathings.

It is well established that a creep test is extremely sensitive to small variations in the product tested and it was recognised that the reproducibility of creep test results obtained from specimens at different parts of a commercial extrusion was not likely to be good. The work now recorded was intended as a preliminary investigation to determine the degree of reproducibility to be expected from materials produced by extrusion on commercial presses and, at the same time, to obtain a general comparison of those materials in use at present.

The work was carried out over a wide range of stresses, in part because of the importance in creep work of the types of ultimate failure, and in part because (in certain practical applications) the general extension obtainable at different stresses, before failure, is of intrinsic interest.

PREVIOUS WORK

Creep

The classical work of Andrade on lead[3, 4] as long ago as 1910 and 1914, showed that creep was made up of two components. His tests, mostly of short duration, were carried out under conditions of constant stress which allowed a mathematical equation to be fitted to the creep curves. One of the constants in this equation, it was suggested, was associated with crystal rotation or crystalline flow, while a second constant represented flow which was of a viscous nature.

In the period 1930–40 Greenwood and his colleagues at the University of Melbourne[5, 6, 7, 8, 9, 10] carried out many creep tests on material prepared by rolling and found that small quantities of alloying elements either

* B.N.F.M.R.A. Report R.R.A. 847P. The work described in this paper was made available to members of the B.N.F.M.R.A. in confidential research reports issued in 1947.

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§ Investigator, B.N.F.M.R.A., London.

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2 McKeown, J., *Engineering*, 1947 **164**, 97.

3 Andrade, E. N. da C., *Proc. Roy. Soc.*, 1910 A, **84**, 1.
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7 Greenwood, J. N. and Worner, H. K., *Proc. Aust. Inst. Min. Met.*, 1936, New Series, No. 104, 385.
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10 Greenwood, J. N. and Orr, C. W., *Proc. Aust. Inst. Min. Met.*, 1939, New Series, No. 113, 1.

increased or decreased the creep resistance of pure lead, depending on the element and the quantity of that element present. On the other hand Gohn, Arnold and Bouton[11], testing specimens of flattened cable sheath, claimed that impurities decreased the resistance to creep at low stresses but increased it at high stresses. In none of this work was an extensive study made of the effect of grain size in creep.

Greenwood and Worner[12] have shown that four distinct types of creep curves may be obtained at low stresses. In the first, strain hardening at the beginning of the test is followed by a marked increase in creep rate owing to recrystallisation. The second type showed an increase in creep rate early in the test and then strain hardening but no recrystallisation: intercrystalline cracking was associated with this type of curve. Type 3 showed a continuously increasing creep rate which was thought to be associated with the loss, during the test, of hardness resulting from previous cold working. Type 4 was the normal strain-hardening variety which is characteristic of metals below the recrystallisation range. These authors have suggested that the limit of proportionality of the material is a guide to a critical stress above which the crystalline component of flow predominates. They consider that this crystalline component of creep is responsible for the recrystallisation associated with curves of Type 1, and that below the critical stress flow is mainly of a viscous nature.

Most of the work reported by Moore, Betty and Dollins[13] from the University of Illinois was carried out on specimens from flattened cable sheath. They concluded that the creep of lead consists of both plastic and viscous flow and were able to present metallographic evidence of viscous flow. As the ratio of these two components varies with stress it was not considered possible to predict the creep properties of lead and its alloys at low stresses from tests of short duration at high stresses. Phillips[14] who tested materials after rolling agreed with this view. Moore, Betty and Dollins[15, 16] gave some evidence that coarse-grained lead had a higher resistance to creep than fine-grained lead.

Hanffstengel and Hanemann[17] found that, with pure lead strip extruded in an experimental press, coarse-grained material was more creep resistant than fine-grained below a critical stress, while the reverse was true above this stress. It was concluded that below the critical stress creep was predominantly of a viscous nature while crystalline flow predominated above the critical stress. A small tensile strain, prior to loading in the creep test, increased the creep rate in all cases, except in coarse-grained lead tested at a stress above the critical point when the creep resistance was increased by the prestrain. Recrystallisation occurring during test greatly increased the rate of creep, a finding in agreement with that of Greenwood and Worner.[12]

A. A. Smith[18] observed that the creep of polycrystalline lead is not necessarily uniform. Testing specimens of strip materials extruded in an experimental press, he showed that, at low stresses, the rate of creep varied abruptly and in an irregular manner throughout the creep test. This was possibly due to the diameter of the grains being equal to the thickness of the strip so that the effect of slip in individual grains could be observed in the creep curve. Moore and his colleagues[15] observed that the creep of single crystals of lead occurred by irregular jumps, although Hirst[19, 20, 21] at the University of Melbourne obtained smooth curves under similar conditions.

Latin[22] recently published the results of internal pressure creep tests, at constant pressure, on cable sheaths of Alloy E (0·4% tin + 0·2% antimony) and pure lead, both materials being in a relatively coarse-grained condition. The circumferential extension at failure decreased with decreasing rate of strain in the case of Alloy E sheaths, while with the pure lead sheaths it was not affected by a change in the rate of strain.

Fatigue

Beckinsale and Waterhouse[25], carrying out tests on a specific stress type of fatigue-testing machine, found that the endurance limit for a specific number of cycles of strain increased with increasing alloying addition. Using a specific strain type of machine Moore and his colleagues[15, 16] confirmed the findings of Beckinsale and Waterhouse and also showed that the endurance limit for a material increased with increasing frequency of cycles of testing. Haigh and Jones,[26] working under conditions of specific stress, found that a grease coating increased the life in fatigue and suggested that this was due to the exclusion of oxygen from the surface of the fatigue specimen. Gough and Sopwith[30] found that the endurance limit of 99·99% lead, under specific stress conditions, was doubled by testing *in vacuo* compared with testing in air. The workers at the University of Illinois[15, 16] showed that the effect of a grease coating increased with increasing number of cycles of strain to failure. Ferguson and Bouton[23] demonstrated that a coating material must be in a fluid form, wetting the fatigue specimen in order to have any effect. Such a coating is highly protective at small deflections and rapid testing rates, but this effect is gradually lost and the coating becomes harmful as the testing speed is reduced or the deflection increased at any one speed.

Metallography

Very little work has been published on the metallography of lead and lead alloys although Gohn, Arnold and Bouton[11] found evidence of discontinuous precipitation at the grain boundaries in plastically deformed antimonial alloys. Similar observations were made by Dean and Ryjord[24] in lead-antimony alloys and in lead-antimony-cadmium alloys. This breakdown of structure followed plastic deformation whether this occurred during a creep or a fatigue test.

MATERIALS TESTED

The range of materials tested, with analyses and mean grain areas, is set out in Table I. In all cases the basic lead was Tadanac of purity 99·997%.

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TABLE I.—ANALYSES AND GRAIN SIZES OF PIPE MATERIALS

Material	Type of Press	Cu %	Ag %	Cd %	As %	Bi %	Sb %	Sn %	Ni %	Zn %	Te %	Tl %	Mean Grain Area mm.²
Tadanac Lead	Pipe	0·0003	0·0003	0·0001	○ 0·0006	0·0002	○ 0·0005	○ 0·0002	○ 0·000025	○ ⩽0·00002	○ 0·0006	Slight trace	0·33
0·005% Silver + 0·005% Copper Alloy	,,	○ 0·005	○ 0·0054	0·0001	n.d.	0·0002	n.d.	n.d.	n.d.	n.d.	n.d.	Slight trace	0·17
0·06% Copper-Lead	,,	○ 0·066	0·0002	⩽0·0001	n.d.	0·0002	n.d.	n.d.	n.d.	n.d.	n.d.	trace	0·005–0·01
Ternary Alloy No. 2	,,	0·0008	0·0007	○ 0·28	n.d.	0·0003	n.d.	○ 1·49	n.d.	n.d.	n.d.	Slight trace	0·001–0·005
1% Silver-Lead	,,	0·0007	○ 0·99	0·0001	n.d.	0·0003	n.d.	n.d.	n.d.	n.d.	n.d.	Slight trace	0·0003
Tadanac Lead	Continuous	0·0002	0·0001	0·0001	○ 0·0006	0·0002	○ 0·0005	○ 0·0002	n.d.	○ ⩽0·00002	○ 0·0006	Slight trace	0·23
0·015% Tellurium Lead	,,	0·0007	0·0003	⩽0·0001	n.d.	0·0007	n.d.	n.d.	n.d.	n.d.	○ 0·014	Slight trace	0·2
Alloy E	,,	0·003	0·0006	0·0001	n.d.	0·001	○ 0·23	○ 0·40	n.d.	n.d.	n.d.	trace	0·06

*These results were obtained by chemical analysis, all other results by spectrographic analysis.
Where an element in the above Table is given as " not detected " (n.d.) the following lower limits of detection in spectrographic analysis should be noted :—

Sn	⩽0·0005 %	Zn	⩽0·001 %
Sb	0·0005 %	Ni	0·001 %
Te	0·001 %	As	0·001 %

The lower limit of detection of Tl is ⩽0·0002%.

The pipes, nominally ¼-in. bore, 6 lb./yd. (0·88 in. outside diameter), were extruded on :—

(i) A standard inverted pipe press.

(ii) A continuous extrusion press of the Pirelli type.

In both machines the pipes were extruded in straight lengths and every care was taken at all stages in subsequent handling to avoid any bending of the pipes. The first 60 ft. of pipe of each material to leave the pipe press were discarded before the samples used in the present work were taken. The continuous extrusion press had been running for a period sufficient to attain uniformity of working conditions before selecting samples.

In neither press was a direct measure of the temperature of extrusion obtained since it was not possible to place a thermocouple in the die. Other measurements indicated that the temperature of extrusion was approximately 300° C. in the case of the continuous press and about 150° C. in the pipe press.

In the continuous machine the speed of extrusion was about 16 ft. per minute and the product was quenched by water jets about 9–12 in. from the die. Extrusion on the pipe press was intermittent to permit the lengths to be cut off. The material from this press was air cooled.

OBSERVATIONS ON STRUCTURES

All the microspecimens examined during the course of this work were prepared in the following manner to ensure that the surface finally examined under the microscope was free from distortion :—

Microspecimens were cut from the pipes with a sharp hacksaw and machined on a lathe by successively finer cuts. Any distorted material remaining from this operation was then removed by alternately polishing with commercial metal polish on Selvyt cloth, and etching in Russell's reagent.[27] The surface finish obtained with metal polish was improved by a final polish on Selvyt cloth with a suspension of levigated magnesium oxide in water. The microspecimens were given a " polish etch " in Worner and Worner's solution[28] followed by an etch in the following solution to reveal the grain boundaries :—

10 grams ammonium molybdate.
25 grams citric acid.
100 cc. water.

The pipes were examined metallographically shortly after extrusion and also after they had been stored for a period of two years at atmospheric temperature.

Structures Shortly after Extrusion

The effect of alloying additions to the Tadanac lead, was to decrease the grain size to an extent depending on the quantity and nature of the alloying element and the temperature of extrusion. The definite difference in the shape of the grains of the material produced on the two types of press was probably due to the different rates of cooling the product. The grains in materials produced on the continuous press were uniform in size and shape, while the vertical pipe press produced material with crystals which were somewhat irregular in shape and varied in size.

None of the materials consisted of a single homogeneous phase. This was to be expected in alloys containing an insoluble phase such as the 0·06% copper alloy and the 1% silver alloy, but not in the Tadanac lead or in the materials where the alloying additions were presumably within the solid solubility limits at the temperature of extrusion.

This heterogeneity was most clearly seen in longitudinal sections. It was not observed in transverse microspecimens of materials produced on the pipe press but the insoluble phase in the 0·06% copper alloy and the 1% silver alloy gave rise to a multitude of black spots distributed uniformly across the cross section. Transverse microspecimens of all pipes produced on the continuous press showed this lack of homogeneity in a characteristic fashion (Fig. 1). In these materials four lines, which were easily seen by the naked eye, divided the microspecimens into quadrants and within each quadrant there was a series of circular lines, somewhat like wood markings in a tree, which were most pronounced and close together near the outside circumferences of the pipes.

Microspecimens taken in the longitudinal direction of all the materials also showed these lines. In the

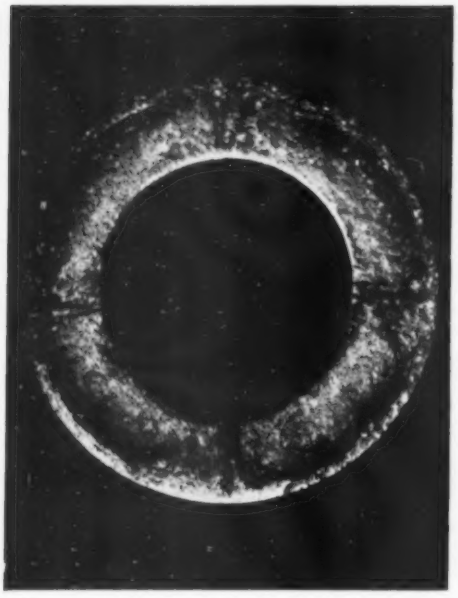


Fig. 1.—Transverse section of Alloy E, produced on continuous press, showing welds and "segregation lines." × 3.

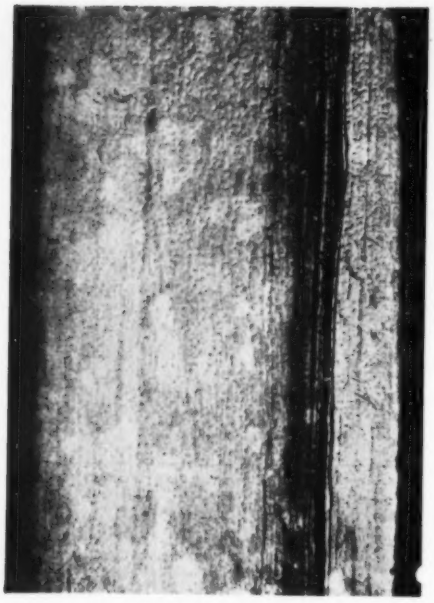


Fig. 2.—Longitudinal section of Alloy E, showing major "segregation line." × 12

materials from the pipe press, including the Tadanac lead, these lines were uniformly distributed across the thickness of the pipe wall but in the materials from the continuous press they were most dense and most closely packed near the outside circumference of the pipes (Fig. 2). Longitudinal microspecimens of the 0·06% copper alloy and the 1% silver alloy, extruded on the pipe press, showed lines, probably consisting of stringers of copper and silver, respectively, uniformly distributed across the wall of the pipes.

Unlike stringers of oxide inclusions, none of these lines in the materials from either press had hindered the process of grain growth after the recrystallisation which occurred during the extrusion process. It is considered that these lines are due to segregation and strong evidence in support of this theory is presented in the appendix. These lines almost certainly indicate the direction of movement of the metal during extrusion, but this is regarded as an effect, rather than a cause and they are, therefore, described as "segregation lines" throughout the rest of this paper.

Structure Two Years after Extrusion

Examination after a period of two years storage at room temperature showed some changes in structure from that immediately after extrusion. In alloy E continuous precipitation* had occurred in the regions where the segregation lines were most dense and he precipitated particles appeared along crystallographic planes while discontinuous precipitation was just beginning and appeared as white lines at the grain boundaries (Fig. 3). In the case of the 0·015% tellurium alloy no continuous precipitation was observed, but broadening of the grain boundaries indicated that discontinuous precipitation had commenced. No metallographic changes were observed in any of the other materials.

* Continuous precipitation occurs when the X-ray diffraction lines gradually move from their original positions, corresponding with the supersaturated solid solution, to new positions corresponding with the equilibrium composition at the ageing temperature. In discontinuous precipitation, the original X-ray lines decrease in intensity and, at the same time, new lines appear, corresponding with the solid solution stable at the ageing temperature, and increase in intensity until the lines of the original solid solution disappear.

Discontinuous precipitation, which occurs by localised decomposition of the supersaturated solid solution, generally starts at grain boundaries and spreads into the grains, whereas the precipitate associated with continuous precipitation occurs along crystallographic planes within the grains.

CREEP TESTS

Testing Procedure

The ends of the pipe specimens were plugged and the load applied through a mechanical form of grip, care being taken to ensure that the loading was axial. All the tests were carried out in a constant-temperature room, at an air temperature of 20° ± 1° C. Extensions were read on a gauge length of 4 in. by means of a measuring microscope sensitive to ± 0·0002 in. (± 0·005%). Lightly scribed marks on the surface, at 0·5 in. intervals, enabled the general and local extensions to be determined after fracture of the specimens. All test specimens were degreased both externally and internally before being set up for test. Considerable care was taken at all stages to avoid any bending or other form of cold working of the specimens prior to loading.

Reproducibility Tests

Reproducibility tests on the materials gave the results in Table II. The reproducibility of creep behaviour in specimens taken from the same length of pipe was found to be reasonably good, but that in specimens taken from different lengths was not so good. The greatest lack of reproducibility was observed in specimens of the Ternary Alloy No. 2 and no explanation was

Fig. 3.—Continuous precipitation within grains and discontinuous precipitation starting at grain boundaries in Alloy E during ageing at room temperature for two years. × 2,000.

found for this. Microspecimens taken from the ends of the creep specimens prior to the start of the tests did not reveal any structural differences which might account for the different creep properties. These microspecimens were also examined by an X-ray technique, similar to that described by C. S. Barrett,[29] which can reveal the presence of cold work in individual crystals. While the grain size of the samples was too fine to be ideal for this technique all the microspecimens appeared to be completely work free. X-ray examination also indicated that there was no difference in lattice parameter between microspecimens, hence it was unlikely that there was more of the alloying elements in solid solution in one specimen than in another. To confirm further that the pipes had not been slightly cold worked by bending, the microspecimens were annealed for 1 hour at 175° C. Although grain growth did occur the absence of localised critical grain growth showed that the pipe had not been cold worked by bending.

Owing to the poor reproducibility of the creep results, great care is necessary in drawing conclusions as to the relative creep properties of the various alloys. However, within the limitations set by the materials as produced, much information of a preliminary character has been obtained, and it is felt that discussion of this may be of assistance in planning future work on creep-resisting lead alloys.

TABLE II.—REPRODUCIBILITY IN CREEP TESTS

Material	Stress lb./in.²	Constant Rate of Creep. Strain/hour	Life Hours	General Extension %
Tadanac lead from pipe press	500	$1{\cdot}15 \times 10^{-5}$	10,800	20 — From same length
	500	$1{\cdot}13 \times 10^{-5}$	11,000	30
	500	$1{\cdot}10 \times 10^{-5}$	11,150	30
	750	$1{\cdot}3 \times 10^{-4}$	650	* — From different lengths
	750	$0{\cdot}96 \times 10^{-4}$	700	20
	750	$1{\cdot}31 \times 10^{-4}$	490	20
	750	$1{\cdot}8 \times 10^{-4}$	348	28
	1,000	Not determined	26	22 — From same length
	1,000	" "	24	22
	1,000	" "	24	22
0·005% Ag + 0·005% Cu Alloy from pipe press	750	$5{\cdot}8 \times 10^{-5}$	2,280	30 — From different lengths
	750	$5{\cdot}4 \times 10^{-5}$	2,760	22
	1,000	$2{\cdot}77 \times 10^{-4}$	544	32 — From same length
	1,000	$3{\cdot}00 \times 10^{-4}$	520	26
	1,000	$2{\cdot}77 \times 10^{-4}$	580	30
0·06% Cu Alloy from pipe press	500	$3{\cdot}25 \times 10^{-5}$	8,700	** — From same length
	500	$3{\cdot}9 \times 10^{-5}$	7,650	**
	500	$5{\cdot}7 \times 10^{-5}$	6,150	80 — From different lengths
	750	$4{\cdot}1$ and $1{\cdot}9 \times 10^{-4}$ ***	1,126	43
	750	$3{\cdot}0$ and $2{\cdot}6 \times 10^{-4}$ ***	1,050	60
	1,000	$2{\cdot}5$ and $1{\cdot}7 \times 10^{-3}$ ***	110	34 — From same length
	1,000	$2{\cdot}0$ and $1{\cdot}1 \times 10^{-3}$ ***	114	34
	1,000	$2{\cdot}2$ and $1{\cdot}2 \times 10^{-3}$ ***	119	34
Ternary Alloy No. 2 from pipe press	500	$8{\cdot}6 \times 10^{-5}$	5,300	225 — From different lengths
	500	$6{\cdot}4 \times 10^{-5}$	5,650	168
	500	—	6,900	158
	500	—	7,750	126
	500	—	—	56 (in 15,000 hrs.)
	750	$2{\cdot}4 \times 10^{-4}$	1,400	140 — From different lengths
	750	$1{\cdot}5 \times 10^{-4}$	1,700	52
	1,000	Not determined	500	80 — From same length
	1,000	" "	500	96
	1,000	" "	500	120
Tadanac lead from continuous press	500	$2{\cdot}3 \times 10^{-5}$	5,600	34 — From same length
	500	$2{\cdot}4 \times 10^{-5}$	6,250	32
	500	$2{\cdot}1 \times 10^{-5}$	6,150	36
	500	$1{\cdot}8 \times 10^{-5}$	6,650	* — From different lengths
	500	$2{\cdot}25 \times 10^{-5}$	5,800	32
	750	$8{\cdot}3 \times 10^{-4}$	110	34
	750	$10{\cdot}0 \times 10^{-4}$	116	40
	1,000	$3{\cdot}1 \times 10^{-3}$	7	50 — From same length
	1,000	$3{\cdot}3 \times 10^{-3}$	6½	72
	1,000	$3{\cdot}1 \times 10^{-3}$	7¼	50
0·015% Te lead from continuous press	1,000	$1{\cdot}24 \times 10^{-3}$	150	35 — From same length
	1,000	$1{\cdot}08 \times 10^{-3}$	180	35
	1,000	$1{\cdot}18 \times 10^{-3}$	150	35
Alloy E from continuous press	1,000	$6{\cdot}2 \times 10^{-4}$	428	44 — From same length
	1,000	$6{\cdot}8 \times 10^{-4}$	390	40
	1,000	$5{\cdot}9 \times 10^{-4}$	478	40

* Gauge lengths not marked for determination of uniform extension.
** Fractured specimens showed a uniform taper from the fracture to the grips, thus making impossible the determination of uniform elongation.
*** Figures given are for creep rates before and after recrystallisation.

Tests on the Materials in the " As-Extruded " Condition

The results of tests at all stresses, including those set out in Table II, are summarised in Table III. Where a number of specimens were tested at one stress in the reproducibility tests, the results given are those for the specimens with the greatest and the least creep resistance. In each material the specimens tested at 300 and 400 lb./sq. in. were taken from the same 6 ft. length of pipe ; a different length was used for the tests at the higher stresses. At the lower stresses some of the tests have not yet proceeded far enough to enable true minimum creep rates to be determined, and the creep rates quoted in Table III are those observed at the present stage of the test. Results are not included for Ternary Alloy No. 2 as the variations shown by this material in the reproducibility tests were considered to be too great to permit of any reliance being placed on the results at different stresses.

It can be seen in Table III that a few of the alloys have higher resistance in creep than the pure leads at low stresses, and at all stresses the material with highest

TABLE III.—RESULTS OF CREEP TESTS

Material	Mean Grain Area mm.²	Stress lb./in.²	Constant Creep Rate Strain/Hour	Life in Hours	General Extension %	Total Extension of Unbroken Specimens %
Tadanac lead from pipe press	0·33	300	$1{\cdot}3 \times 10^{-6}$	22,000 U		3·37
		400	$3{\cdot}9 \times 10^{-6}$	22,000 U		12·50
		500	$1{\cdot}1$–$1{\cdot}15 \times 10^{-5}$	10,800–11,150 B	20–30	
		750	$9{\cdot}6$–$18{\cdot}0 \times 10^{-5}$	348–700 B	20–28	
		1,000	—	24–26 B	17–22	
0·005% Ag + 0·005% Cu Alloy from pipe press	0·17	300	$3{\cdot}0 \times 10^{-7}$	22,000 U		1·03
		400	$2{\cdot}4 \times 10^{-6}$	22,000 U		6·28
		500	$3{\cdot}2 \times 10^{-6}$	22,000 U		10·25
		750	$5{\cdot}4$–$5{\cdot}8 \times 10^{-5}$	2,280–2,760 B	22–30	
		1,000	$2{\cdot}8$–$3{\cdot}0 \times 10^{-4}$	520–580 B	26–30	
0·06% Cu Alloy from pipe press	0·005–0·01	300	$5{\cdot}3 \times 10^{-6}$	22,000 U		14·30
		400	$1{\cdot}4 \times 10^{-5}$	22,000 U		39·0
		500	$3{\cdot}3$–$5{\cdot}7 \times 10^{-5}$	6,150–8,700 B	80	
		750	$2{\cdot}3$–$4{\cdot}1 \times 10^{-4}$ *	1,050–1,126 B	43–60	
		1,000	$1{\cdot}3$–$2{\cdot}2 \times 10^{-3}$ *	110–120 B	23–34	
1% Ag Alloy from pipe press	0·0003	300	$8{\cdot}6 \times 10^{-6}$	20,000 U		0·33
		400	$4{\cdot}8 \times 10^{-7}$	20,000 U		1·16
		500	$1{\cdot}2 \times 10^{-6}$	20,000 U		2·73
		750	$9{\cdot}3 \times 10^{-6}$	20,000 U		34·4
		1,000	$2{\cdot}4 \times 10^{-5}$	12,050 B	60–88	
Tadanac lead from continuous press	0·23	300	$1{\cdot}9 \times 10^{-6}$	22,000 U		5·45
		400	$5{\cdot}0 \times 10^{-6}$	22,000 U		20·94
		500	$1{\cdot}8$–$2{\cdot}4 \times 10^{-5}$	5,600–6,650 B	24–32	
		750	$0{\cdot}8$–$1{\cdot}0 \times 10^{-3}$	110–116 B	34	
		1,000	$3{\cdot}1$–$3{\cdot}3 \times 10^{-3}$	6¾–7¼ B	50–72	
0·015% Te lead from continuous press	0·20	300	$1{\cdot}6 \times 10^{-7}$	17,000 U		0·40
		400	$3{\cdot}3 \times 10^{-7}$	22,000 U		1·35
		500	$1{\cdot}2 \times 10^{-6}$	22,000 U		5·10
		750	$6{\cdot}4 \times 10^{-5}$	1,512 B	20	
		1,000	$1{\cdot}1$–$1{\cdot}2 \times 10^{-3}$	150–180 B	35	
Alloy E from continuous press	0·06	300	$6{\cdot}8 \times 10^{-7}$	22,000 U		1·66
		400	$1{\cdot}7 \times 10^{-6}$	22,000 U		4·32
		500	$5{\cdot}0 \times 10^{-6}$	19,390 B	14	
		750	$1{\cdot}1 \times 10^{-4}$	1,680 B	28	
		1,000	$5{\cdot}9$–$6{\cdot}8 \times 10^{-4}$	390–440 B	37–44	

* Figures given are averages for creep rates before and after recrystallisation.
U = Unbroken. B = Broken.

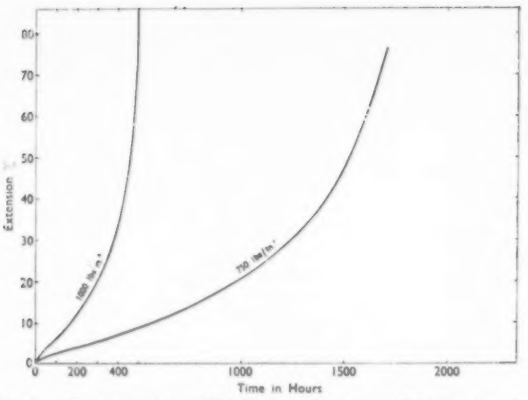


Fig. 4.—Creep tests at 750 lb./sq. in. and 1,000 lb./sq. in. on Ternary Alloy No. 2.

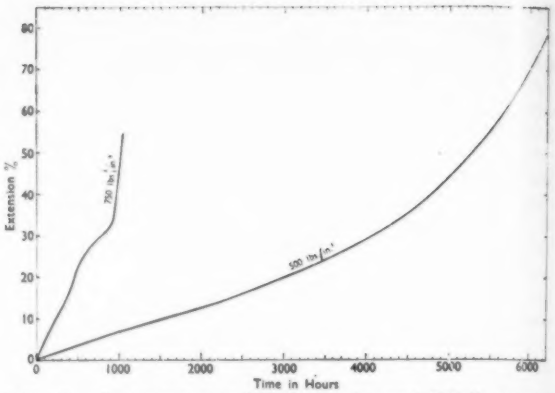


Fig. 5.—Creep tests at 500 lb./sq. in. and 750 lb./sq. in. on 0·06% Cu lead.

resistance to creep is the 1% silver alloy. The 0·005% Ag + 0·005% Cu alloy showed remarkable resistance to creep for a material with such low alloying additions; its grain size, however, was large and comparable with that of pure lead. The good creep resistance of the 0·015% tellurium lead at low stresses was noteworthy and this material also had a large grain size.

Types of Creep Curves Obtained

All the materials with the exception of the Ternary Alloy No. 2 and the 0·06% copper alloy gave a normal strain-hardening type of creep curve. The curves for Ternary Alloy No. 2 (Fig. 4) showed a small amount of strain hardening early in the test, and then a continuously increasing creep rate. In Greenwood and Worner's analysis such curves were classified as Type 3 and the specimen was considered to be losing hardness conferred by previous cold working. This does not agree with the findings on the present material which, as noted earlier, had been shown by X-ray and metallographic examination to be sensibly work free. Creep curves at 500 and 750 lb./sq. in. obtained with the 0·06% copper lead are shown in Fig. 5. The curve at 500 lb./sq. in. is of the Type 3 in the Greenwood and Worner classification, while the curve at the higher stress is of Type 1 and shows strain hardening at the beginning of the test, followed by a marked increase in rate of creep due to recrystallisation.

Metallographic Examination of Specimens after Test.

A. Materials Produced on the Pipe Press

(i) *The Tadanac Lead.*—This Tadanac lead, when tested at a stress of 500 lb./sq. in., failed with very little local ductility. Extensive intercrystalline cracking was present and some recrystallisation may also have occurred. At stresses of 750 lb./sq. in. and above there was no evidence of intercrystalline cracking, but recrystallisation was extensive and produced irregular structures of a grain size similar to that of the material in the as-extruded state.

(ii) *The 0·005% Ag + 0·005% Cu Alloy.*—When tested at stresses of 750 and 1,000 lb./sq. in. this material showed intercrystalline cracking which was less extensive at the higher stress. Recrystallisation occurred at both these stresses, with rather more at the higher stress. At stresses of 1,250 lb./sq. in. and above, the local ductility at fracture increased and no intercrystalline cracking was observed. Recrystallisation probably occurred during the tests at these high stresses and the resulting grain size was a little smaller with increasing stress. Presumably the alloying elements were in solid solution at 20° C. as no precipitation was observed after test at any stress.

(iii) *The 0·06% Copper Alloy.*—After test at a stress of 500 lb./sq. in. the specimens showed intercrystalline cracking and had recrystallised to a fine grain size of the same order as the material in the as-extruded state. At all the higher stresses no intercrystalline cracking occurred and recrystallisation resulted in a grain size much larger than in the material before test. No precipitation was observed in this material after test at any stress. Although precipitation possibly occurred during the tests, as the result of recrystallisation of the supersaturated matrix in equilibrium at the temperature of extrusion, it is probable that such a precipitate, if it occurred, was not detected owing to the presence in the alloy of free copper in excess of the solid solubility at the temperature of extrusion.

(iv) *The Ternary Alloy No. 2.*—After test at stresses of 500, 750 and 1,000 lb./sq. in. this material showed intercrystalline cracking to a small extent which was absent at higher stresses. Recrystallisation probably occurred at all stresses and the mottled structures obtained after test were

Fig. 6.—Recrystallisation and discontinuous precipitation at the grain boundaries, and strain lines within the grains in the 0·015% tellurium alloy after failure at a stress of 1,500 lb./sq. in. × 100.

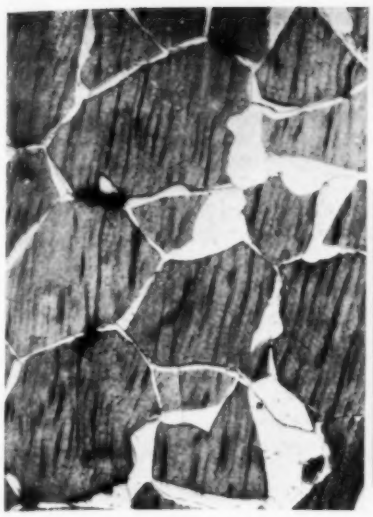


Fig. 7.—Discontinuous precipitation, chiefly of the "band type," at the grain boundaries in a specimen of Alloy E after failure at a stress of 500 lb./sq. in. × 75.

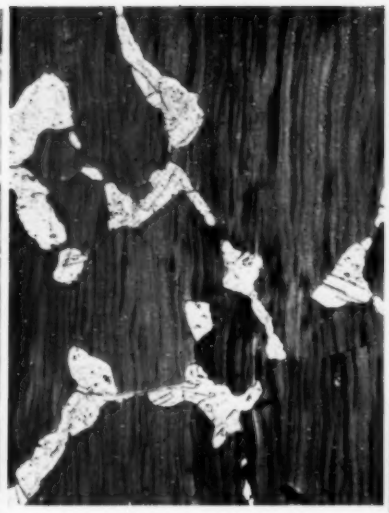


Fig. 8.—Discontinuous precipitation of the "recrystallisation type" at the boundaries in a specimen of Alloy E after failure at a stress of 2,000 lb./sq. in. × 75.

Fig. 9.—Discontinuous precipitation of both the "recrystallisation" and "unstable" types in a specimen of Alloy E after failure at a stress of 2,000 lb./sq. in. × 250.

probably indicative of a small quantity of globular precipitate. No progressive discontinuous precipitation was observed in failed specimens tested in the as-extruded condition, although it occurred during creep in specimens of this alloy which had been annealed for 1 hour at 250° C.

(v) *The* 1% *Silver Alloy.*—As yet, failures in this material have only been obtained in tests at very high stresses. The fractures were of the ductile, knife-edge type, with no intercrystalline cracking. Recrystallisation occurred near the point of failure, as the grain size diminished towards the fracture. The parts of the specimen well removed from the fracture may also have recrystallised, but this was not made obvious by a change in grain size. No decomposition of the matrix of the alloy was observed, although, as in the case of the 0·06% copper alloy, it may have occurred.

B. Materials Produced on the Continuous Press

(i) *Tadanac Lead.*—Tadanac lead, when tested at all stresses between 500 and 1,250 lb./sq. in., failed in a ductile manner. Recrystallisation occurred during the tests at these stresses, producing a coarse-grained structure after testing at stresses of 500 and 750 lb./sq. in., and a structure of finer grain size than in the original material at stresses of 1,000 and 1,250 lb./sq. in. Intercrystalline cracking was completely absent after test at all stresses except in the specimen tested at 500 lb./sq. in., where the extent of cracking was very slight.

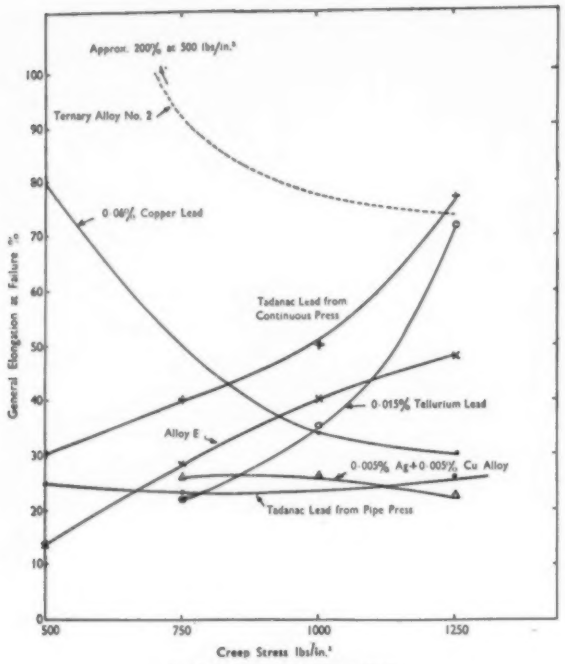
(ii) *The* 0·015% *Tellurium Alloy.*—This alloy was very resistant to recrystallisation during creep tests at all stresses. After failure at the highest stresses there was only a small number of recrystallised grains in the grain boundaries of the original grains even in the region of the fracture (Fig. 6). The specimens tested at stresses up to 1,250 lb./sq. in. showed extensive intercrystalline cracking which tended to diminish with increasing stress. At stresses greater than 1,250 lb./sq. in. intercrystalline cracking was entirely absent.

After failure at the stress of 750 lb./sq. in. there was no evidence of recrystallisation but the grain boundaries were broad, indicating that discontinuous precipitation was occurring there. Within the original grain there was a cellular network which suggested that continuous precipitation had occurred in the richest portions of the cored structure remaining after extrusion. Creep at a stress of 1,000 lb./sq. in. produced one or two recrystallised grains and discontinuous precipitation at the grain boundaries of the original crystals; the cellular network was again present. The discontinuous precipitation appeared at all the original grain boundaries, but extended into only one of the parent grains as a narrow band of new material, enclosed on one side by a ragged boundary and on the other by a straight line. Specimens tested at stresses of 1,250 lb./sq. in., and above, similarly showed discontinuous precipitation at the grain boundaries and the number of recrystallised grains increased slightly with increasing stress. At stresses of 1,500 and 1,750 lb./sq. in. the cellular network of continuous precipitation within the original grains was replaced by precipitation occurring as fine lines which were probably associated with slip planes resulting from the excessive deformation (Fig. 6).

(iii) *Alloy E.*—Specimens of Alloy E tested at stresses of 500, 750 and 1,000 lb./sq. in. gave a fracture with little local ductility, but at stresses of 1,750 lb./sq. in. and above the fracture was of the ductile, knife-edge type. Intercrystalline cracking was only present in specimens which failed with the former type of fracture. Generally, when intercrystalline cracking occurred it was in the region of the major segregation lines, near the outside circumference of the pipe. In some cases a longitudinal crack was observed following a major segregation line for a considerable distance.

Continuous precipitation was present in the material before test and, owing to difficulties of resolution, it is not possible to say whether or not this type of precipitation increased during tests at stresses up to 2,500 lb./sq. in.

Discontinuous precipitation occurred during creep tests at all stresses. For the sake of clarity the three



GRAIN SIZE OF MATERIALS.

Material	Press	Mean Grain Area Mm.²
Tadanac Lead	Pipe	0.33
0.005% Ag + 0.005% Cu Alloy...	,,	0.17
0.06% Cu Alloy	,,	0.005—0.010
Ternary Alloy, No. 2	,,	0.001—0.005
Tadanac Lead	Continuous	0.23
0.015% Te Alloy	,,	0.20
Alloy E	,,	0.06

Fig. 10.—General elongation—creep stress curves.

different types of this precipitation will be referred to as (*a*) the band type ; (*b*) the recrystallisation type ; and (*c*) the unstable type :—

(*a*) This occurred as a narrow band at the grain boundaries (Fig. 7) in a very similar fashion to that observed in the 0·015% tellurium alloy. In the case of Alloy E, this band of new solid solution was often wider and more distinct than in the tellurium alloy. Only an occasional grain boundary could be seen within this band and often none could be observed.

(*b*) The recrystallisation type of discontinuous precipitation appeared at localised points along the grain boundaries instead of as a band. Frequently only one of the adjacent grains had partly decomposed, forming a new solid solution with only one orientation, and which had the appearance typical of a recrystallised grain growing into one of its parent grains. Sometimes, the new solid solution, instead of being of only one orientation, consisted of a number of small crystallites, the boundaries of which were easily observed. At other times the new solid solution grew from localised points at the grain boundaries into both parent grains at the same time (Fig. 8) and gave the impression of true recrystallisation.

(*c*) Normally, the new solid solution formed by discontinuous precipitation, being depleted in antimony, etched white as compared with the dark shade of brown of the original grains. At some grain boundaries, however, discontinuous precipitation occurred as in the " band type " but for the most part etched the same shade of brown as the original grains. Within the boundaries of these areas of discontinuous precipitation, however, the antimony-rich constituent could be seen along crystallographic planes (Fig. 9). The region immediately behind the advancing boundary of the precipitation in this photomicrograph etched dark but includes a number of light spots.

Specimens tested at stresses of 500 and 750 lb./sq. in. showed the " band type " of precipitation, but the band was not so uniform in width as in the 0·015% tellurium alloy. The recrystallisation type was also present to a limited extent and confined to the form in which decomposition occurred in only one of the parent grains constituting a grain boundary. The unstable type of precipitation was completely absent. With specimens tested at higher stresses, the band type of precipitation decreased in quantity while the recrystallisation and the unstable types increased in quantity with increasing stress.

These two latter types were associated chiefly with the major segregation lines. At the highest stresses the recrystallisation type became characteristic of a partly recrystallised material as the new solid solution grew from localised points in a grain boundary into both the adjacent original grains.

Variation of General Elongation with Creep Stress

The above comparison of the materials has been made on the basis of minimum creep rate. Another important factor in the creep behaviour is the extension to failure. Such extension may be divided into two parts, general and local, and of these the more important is the general extension. Once local extension begins the life of a creep specimen is virtually at an end.

The general elongation of those specimens which have failed in the creep tests is plotted against the creep stress in Fig. 10.

For stresses between 500 and 1,250 lb./sq. in. the curves obtained fall into three groups :—

1. The general elongation falls with increase in creep stress.
2. The general elongation rises with increase in creep stress.
3. The general elongation remains constant with change in creep stress.

In attempting to fit the materials into this grouping, it is necessary to consider those from the pipe press separately from those from the continuous press. Also the metallographic examinations of the fractured specimens showed that intercrystalline cracking at low stresses was more pronounced in some of the materials than others and the difference in this respect probably contributed to difference in general elongation.

Of the materials extruded on the pipe press, the fine-grained ones gave slight intercrystalline cracking at low stresses and no intercrystalline cracking at high stresses, while the general elongation fell with increase in creep stress. The coarse-grained ones gave considerable intercrystalline cracking at low stresses and little or none at high stresses, while the general elongation remained constant with change in creep stress.

All three materials extruded on the continuous press showed a rise in general elongation with increase in creep stress. In the two alloys considerable intercrystalline cracking occurred in the tests at low stresses and some was also found at the highest stresses, whereas in the Tadanac lead intercrystalline cracking was slight

at the lowest stress and absent at higher stresses. The range of grain sizes in the continuous press materials was too limited to permit any correlation between grain size and intercrystalline cracking. It is of interest to record that some Alloy E strip which had been extruded on a small experimental press, not of the continuous type, and which had a grain size of 0·001 mm.^{2} showed, in creep tests, a fall in general elongation with increase in stress and virtually no intercrystalline cracking even at the lowest stress.

More work is clearly necessary before any conclusions can be reached on the factors responsible for the change in general elongation with change of creep stress. Such work should be carried out on a wide range of grain sizes in any one alloy extruded in one type of press.

[In the concluding part of this article, to be published in the February issue, the authors will deal with the effects of annealing and cold-work on the creep properties and with the results obtained in fatigue testing.]

Costing and Modern Accounting Methods in the Metal Industries

II.—Control of Stores and Metal Stocks

By S. Howard Withey, F.Comm.A.

In many works materials and components are purchased in bulk for stock. It then becomes essential to have an efficient system of storekeeping in order that the stock may always be maintained at such a level that no interruption of production occurs and that the material costs may be correctly allocated to the various orders. In this second article of the series, the author deals with some of the points concerned.

WHERE possible, separate stores should be provided for materials of a bulky nature, the use of which is confined to particular classes of operatives, and when such stores are placed in charge of a foreman he will usually have the keys in his possession and will be responsible for the keeping of accurate records of everything received from suppliers and issued to production.

Weak spots in administration can often be revealed by maintaining a suitable system of stores accounts, and when the stock under a particular heading is getting low it becomes advisable to enter details in a special requisition book, together with a description of the plant and equipment to be used. When stores are obtained by tender, a list is usually made out for inclusion in the tender form, appropriate clauses and conditions also being inserted in regard to such matters as the date of payment of accounts, the package charges, etc.

Purchase of Stores

Normally, most of the stores which are regularly used can be purchased by contract, but the prevailing difficulties experienced in securing supplies at the right time have largely neutralised the advantages of the contract method. Consequently, in practice, the control of stores in the metal industries now resolves itself into the adoption of suitable methods of placing orders, accepting and checking delivery, keeping separate stores accounts, classifying the trade purchases under definite headings and compiling inventories at regular intervals.

While purchases of materials have to be governed to some extent by the current market prices, the special local circumstances are often a more important factor. Current requirements have to be considered in conjunction with the state of the order book and suppliers should be advised that any material deviation from the particulars as enumerated on the order forms will be entirely at their own risk, unless alternative supplies have been specified or other variations authorised. When dealing with buying agents for the first time, the terms and conditions of business should be clearly stated or confirmed, and it is often advisable to specify the prices of materials ordered, the latest date for delivery, and other conditions.

Storekeepers should be quite satisfied as to both quantity and quality before taking stores into stock and, to guard against the possibility of certain descriptions being accepted without sufficient verification, some departmental managers now make a practice of cutting the carbon copies of the order forms in such a way as to remove all references to quantities and prices. The storekeeper is then compelled to subject the consignments to proper examination before making the necessary

STORES ORDER BOOK

Date	Requirements	Quantity					Order Authorised by	Date of Order	Supplier	Date Received	Inwards Book	Ref.

STORES RECEIVED BOOK

Date of Delivery	Order No.	Name and Address of Supplier	Description of Stores Received	Number, Weight or Quantity Received	Rate or Price	Cost Price	Stores Order Book Folio

entry in the inwards book kept in the receiving department.

The initials of the person authorising the placing of the order should be inserted against the entry made in the stores order book, and for office use a separate section should be provided, comprising columns for recording the date; the name and address of the supplier; the date on which the stores actually came to hand; and the page or folio on which the entry appears in the inwards book. A typical ruling is indicated on page 143.

Receipt of Stores

As the receiving, checking and approving are not usually the responsibility of the same person, the invoices or other debiting documents should be stamped by means of a rubber stamp or other suitable impression on the following lines:—

INVOICE STAMP

No. Date 19
Stores Received by
Quantity Verified by
Prices and Discounts Checked by
Taken into Stock by
Passed for Payment by
Inwards Book Ref.
Purchases Journal Folio

The Stores Received Book should give all essential details and is usually provided by the printer with separate columns or sections for the insertion of the date on which the stores arrived on the premises; the order number; name of consignor; the rate or cost price of each different grade or description; and the folio of the stores order book, as indicated above.

Details relating to orders received for the supply of consumable stores are sometimes set down on forms which can be filed until such time as the stores have actually been despatched, when the documents are either placed on another file or bound in book form, and many metal manufacturers prefer to use a duplicate book, the original sheets being handed to the persons or transferred to the departments immediately concerned and the carbon copies retained to serve as the foundation for the entries to be made in the sales book.

Issue of Stores

Each requisition will go to the stores and also to the costing department, the progress slip for the first operation being passed to the appropriate department for entry on the progress card. Particulars relating to stores issued to production may be entered in a book ruled with columns for showing the date of issue; the requisition number; name of department; a description of the stores issued, including the number, weight or quantity; and the invoiced cost (see below).

Parts which have been manufactured and put into stock for use in assembly can now be trucked at the lowest possible cost when issued against requisitions, and experience has proved that when compared with hand-trucking the power-trucking system is an extremely economical method of handling metal parts. Where standard components are manufactured for stock, it is the practice to manufacture under a stock order number and to stock under a stores code, the former being necessary in order to ascertain the cost and the latter to identify the component, and for pricing purposes. The main objects of coding are to assist the storekeeper in storing the components and locating them when needed, to aid the technical office in specifying the components to be used, and to help the costing assistants or ledger section in pricing. A common form of stores code consists of two groups of figures divided by an oblique line, the first figures indicating the group and the second the number of the bin or place in which the component is stored. Group numbers are sometimes employed to indicate the shape, the nature of the material, the state of production, the type of product on which the component is to be used or the location of the stores, and when all the costs for one material are filed together the quantity over a given period can be readily ascertained. In practice, this may have the effect of avoiding the ordering of uneconomic quantities and where possible it is advisable to use the code numbers as the drawing numbers.

In order to find certain stores, operatives often have to spend a good deal of time in searching through boxes crammed with a mass of miscellaneous material, and while some firms incur considerable expense in the

STORES ISSUED BOOK

Date of Issue	Requis. No.	Issued to	Description of Stores Issued	Number, Weight or Quantity Issued	Rate or Price	Cost Price

installation of first class machines, the expedients tolerated for the storage of packing and jointing materials are not always satisfactory. By using racks made of lengths of 2-in. angle iron about two feet long, and attaching one side of the iron permanently to a near-by wall or other convenient structure and slotting the other side to suit different diameters, spare parts can be suspended by the head and the choosing of suitable sizes is then greatly simplified. Racks of a similar type may be supplied to the various departments and it is often advantageous to provide individual machines with their own special racks, as apart from immediately disclosing faulty items, foremen and works' managers are better able to tell the condition and quantity of the supplies in use.

Costing

Orders executed on a contract basis necessitate the keeping of separate accounts on the debit side of which will appear the labour costs, the materials and stores consumed, the equipment used and an adequate sum to cover the overhead charges, and in certain sections of the metal industry dockets are now being used rather extensively and are usually printed in sets of four. Each form should be of distinct colour so that one can be used on the spot, another in the workshop, one in the stores and an authentic copy retained for office use. Such dockets should be numbered in strict rotation, all forms of the same set bearing identical numbers and showing the date and job number and, directly all forms making up a set have been collected in the office, the date of completion should be inserted and summaries compiled giving the time spent and the stores used.

By using an accounting machine which is equipped with totalisers, a separate order form and ledger card can be made out for each order, the form being folded round the card so that the order number can be set down in the appropriate column and the quantity ordered posted in oblique figures in a " Balance Order " column. When this method is adopted, a receipt note bearing the essential details can be passed to the operator for posting purposes, enabling the respective order statement to be selected and inserted in the machine, together with the ledger cards, and the receipts items recorded on both documents at one operation.

Bulk Purchases

With the object of ensuring the delivery of stocks on definite dates some metal traders make a practice of placing bulk orders with a number of suppliers or purchasing agents for the main materials and stores but, under the conditions that now prevail, this does not always result in deliveries in bulk, and unless the data required to complete instruction notes is readily available it is practically impossible to exercise an effective internal check and control over the orders which are outstanding in respect of different descriptions of material.

This difficulty can be overcome by the use of stock ledger cards which are divided into three sections for " Individual Orders," " Outstanding Orders " and " Stock." The order forms should then be provided with columns for recording the pick-up ; order number ; receipts ; balance of order ; and date, and the " Individual Orders " section of the ledger cards should be ruled to correspond. Upon the receipt of stock forming part of a bulk order the item would appear in the " Receipts " column of the order form and the ledger card and would go into the " Balance Order " column in the form of subtraction from the pick-up. This can best be demonstrated by giving a few imaginary transactions involving the placing of orders and the receipt and issue of stock, followed by the entries on the stock ledger card below, thus :—

December 12th.—Placed order (No. 5227) for 300 gross of Drop Handles, size 1½ in.

December 12th.—Ordered 150 gross of 1½-in. Drop Handles (Order No. 5249).

December 17th.—Received 100 gross (5227) and issued 50 gross.

December 19th.—Received 75 gross (5249).

December 20th.—Issued 25 gross (5249).

December 21st.—Received 100 gross (5227).

Although the receipt of stock affects all three sections of the ledger account, only the stock section is affected when stock is issued and it is a simple matter to enter the items and extend the new balances. If the order forms and ledger cards are housed in a separate vertical and visible pocket, references to any of the sections are materially facilitated and a trial balance can be produced each day, with carbon copies to enable the departmental managers to see the current stock position. Attention can be directed to anything unusual by employing signals on the visible edges.

By means of a file of perforated cards a constant and effective control can be maintained over inventories, providing the perforations are made to represent specific values and descriptions. Whenever materials or stores are received from suppliers, the item cards should be placed in appropriate positions, and on the withdrawal

STOCK LEDGER CARD

Individual Orders				Outstanding Orders			Stock		
Article : DROP HANDLE 1½ in.									
Pick-up	Order No.	Receipts	Balance Order	Date	Pick-up	Balance	Pick-up	Issues	Balance
	5227		300	12/12		300			
	5249		150	12/12	300	450			
300	5227	100	200	17/12	450	350			100
				17/12			100	50	50
150	5249	75	75	19/12	350	275	50		125
				20/12			125	25	100
200	5227	100	100	21/12	275	175	100		200

of anything from stock the cards will serve for the preparation of debit notes which can be produced by a tabulator without the necessity of calculating or checking the price extensions. The same cards can also be tabulated to provide detailed analyses of materials, stores, goods and articles received, and the file will always give visual indication of stocks on hand and will provide a complete inventory, automatically priced and valued.

In addition to the outlay in the form of materials, stores and parts and the wages paid to workers, charge hands, foremen, testers, and technical and other assistants, all expenses incurred in the process of manufacture must be covered before any real profit can be earned. In the metal industries, the term " prime cost " usually refers to the cost of materials and labour and such direct expenses as the salaries paid to inspectors and superintendents, the rent of the mill or workshops, expenditure involved in the maintenance of plant, machinery and other wasting assets employed, and depreciation in the value of the productive assets, and the next article in this series will deal with the compilation of mill prime costs, including tube mill and wire mill costs.

The Nickel Industry in 1949

TOTAL world consumption of Canadian nickel in all forms for the year 1949 is expected to be about 15% lower than in 1948, when a new high peace-time record was established. During the early part of the year nickel sales remained high and were comparable to the volumes achieved in the previous year. A sharp drop in the period May through August, however, was followed by improvement during the remainder of the year to date, despite the fact that consumption was adversely affected in the autumn months by the steel and coal strikes in the United States. The fall in demand for various metals, including nickel, required a reduction of nickel production by International Nickel bringing the rate of the last half of the year to approximately 15% below the previous level.

French production of nickel showed an increase over the preceding year, although it was still relatively small as compared with Canadian. Most of this output comes from mines in New Caledonia. The Cuban mines remained closed and no production was reported from that country. Russia continued to produce nickel, but, as has been the case for years, no information was made available on its output of the metal.

The United States again was the largest consumer of Canadian nickel, with approximately 65% of the total being used in that country. Consumption in the United Kingdom was about 20%. These two countries with Canada accounted for approximately 88% of consumption. The remainder went to other countries throughout the world. The United States price of nickel continued unchanged throughout the year. Other world prices were adjusted at the time of the currency devaluations in September to retain parity with United States prices.

International Nickel continued this year its extensive plant programme in Canada designed to improve production methods, to reduce costs and to make use of the great reserves of low-grade ore which heretofore had been deemed unprofitable.

The very broad diversification of uses of nickel and its alloys that have been successfully developed in the industrial world and the wide range of their properties continue to give users confidence and ensure the maintenance of consumption. While the consumption in low alloy steels was considerable during the year, progress was especially noticeable in the higher alloyed materials, particularly in the use of nickel-chromium stainless steels. In addition to the motor-car industry and equipment for other forms of transport, it is noteworthy that the building field has become an important user of these steels for structural and architectural components which are exposed to the elements. In the manufacture, transportation and handling of chemicals, drugs, food products and beverages, large quantities of this steel continued to be employed. Household appliances, restaurant equipment, shipping containers, sanitary equipment and a multitude of various items continued to be manufactured in these stainless steels.

An interesting development which has accounted for considerable consumption of stainless steels has been their wider employment for lining pulp digesters in paper manufacture. The new processes for treating fats and oils for the soap and detergent industries also are requiring extensive quantities of these steels for processing equipment operating at high temperatures and pressures. The ability of chromium-nickel stainless steels to maintain their strength and toughness at subzero and liquid air temperatures has prompted their use in many other varied fields.

In this country and on the Continent, consumption of stainless steels has been held back by insufficient production facilities. However, further increases in the applications of these steels are expected when additional fabricating equipment, primarily rolling mills, is available. The major demand for stainless steels was from the oil and chemical industries which, particularly in Britain and France, are being greatly expanded. Fruit and processing fields, including milk handling, consumed large quantities of these steels in Britain, and experiments are now under way for their use in the construction of barges used to transport coal. The ceramic industry in France is employing stainless-steel vats for handling special grades of clay which require treatment under difficult conditions.

Although particular attention has been directed to the use of nickel-chromium stainless steels, applications of high-nickel alloys in such fields as power, railway and marine engineering were equal in importance with those which have developed in the chemical groups, where Monel, nickel and Inconel are increasingly used. In power-plant equipment, Monel and " K " Monel in rod form led the requirements with cast Monel, cast " S " Monel and Inconel also in demand. Uses ranged from valve trim, especially in handling high-pressure steam, to tubing for both high and low pressure preheaters. The railway field turned to the use of Monel, especially for fresh water storage tanks on passenger cars. Lower maintenance costs and elimination of rusty water were primarily responsible for this application. A newly-designed evaporator for producing fresh water from salt water came into wide use during the year. This evaporator performed so successfully on shipboard that it has been introduced to the processing and chemical industries for other types of distillation. It is made entirely of Monel sheet.

The Corrosion of the Austenitic Stainless Steels

Part I—Types of Stainless Steel and Forms of Attack

By G. T. Colegate, B.Sc., A.I.M.

Considerable progress has been made, during the last thirty years, in the development and application of the austenitic stainless steels. In a series of four articles, the author discusses many factors governing the corrosion of these steels, including the effect of alloying elements in addition to chromium and nickel, and of the corrosive media with which the material may come into contact in service.

ALLOYS of chromium and iron, nickel and iron and of all three elements were known and used towards the end of the 19th century, but the additions of nickel or chromium or both were then only made to steel for the purpose of improving mechanical properties. The use of such alloys was gradually extended up to the beginning of the first world war, and it was about that time that Brearley was attempting to develop a steel suitable for use as a gun-barrel material to resist fouling and erosion. One of the steels tested was a material containing about 13% of chromium and during metallurgical examinations of this material it was found to be exceptionally resistant to acid etching. This suggested to Brearley its possible use as a stainless alloy in such applications as cutlery manufacture, for which it was subsequently tried. In due course, Brearley took out a patent covering steels with a chromium content of from 9–16% and with a carbon content of 0·70% maximum. Commercial manufacture of this class of steel started in 1914.

Much of the development on the austenitic stainless steels was carried out by the Krupp concern in Germany beginning about 1909 and was largely the result of some work which was being carried out by them to develop new thermo-couple alloys. The starting point in this case was the group of nickel-iron alloys which by that time were fairly well known, and it was reasoned that addition of chromium to such alloys might well confer upon them some of its own resistance to oxidation. Once again, it was during the routine laboratory examination of the series of alloys produced that it was noticed that those materials which contained 20% of chromium remained uncorroded in the laboratory atmosphere. The significance of this was at once realised and the corrosion resistance of the materials towards natural waters, including sea-water, was investigated. Finally, an alloy containing 20% chromium and 7% nickel was developed and was patented in 1912. Further development work indicated that the 18% chromium, 8% nickel alloy was the best all-round alloy in the austenitic class.

Meanwhile, alongside all this work and in conjunction with it, was proceeding the development of other types of alloy such as the heat-resistant ones, which are, however, outside the scope of this article.

It is intended in this short series of articles to consider various aspects of the corrosion resistance of one particular group of stainless steels—namely, the austenitic nickel-chromium alloys. These are probably the best known group of industrial alloys (the famous 18 : 8 composition being a typical representative of this class), although, of course, many alloys from other groups of stainless steels are widely used—e.g., ordinary cutlery steel which is usually of the martensitic type. To deal fully with the whole subject of the corrosion of the stainless steels would require a book, so extensive has the number of these alloys now become and so wide is the field of their industrial application. The chemical, food, petroleum and transport industries are only a few of the more important users who have found extensive applications for various stainless steels and who, in many instances, have developed alloys specially for their own use.

Types of Stainless Steel

The stainless steels may be divided into three main groups, all of which are available in both wrought and cast forms. The first group is that containing the martensitic chromium steels. These are essentially alloys of iron and chromium containing between about 12 and 18% of the latter element. These steels are hardenable and may be modified by the addition of nickel or other elements, although, at least in the case of the nickel, these additions do not appreciably influence the corrosion properties of the alloy, but are introduced in order to improve heat-treatment properties, working properties, etc. This type of steel is used in cutlery and in many other applications where the exposure is not particularly severe.

The second group of stainless steels are the ferritic ones containing between about 14 and 30% of chromium. Unlike the martensitic group, these steels are virtually non-hardenable. Their higher chromium content confers very much greater resistance to corrosion on them and, in particular, resistance to high-temperature oxidation.

It is with the third group of stainless steels that we are concerned, namely, the austenitic ones. Their composition varies from about 16–25% of chromium with, in addition, about 6–22% nickel. Like the ferritic steels, the austenitic group are non-hardenable by heat-treatment, though they will work-harden. They have good working properties and higher strength than the straight chromium steels and are easily fabricated by welding. All these factors, combined with excellent corrosion resistance, have resulted in this class of steel being the most widely used of the stainless steels.

Not only is it proposed to limit the series to one group of alloys, but it is also intended to omit any discussion of the high-temperature corrosion properties of the stainless steels since, although resistance to high-temperature oxidation and to ordinary aqueous or atmospheric environments are both dependent on very

much the same factors, the resistance to high temperatures is, from the practical application point of view, a subject that is best dealt with on its own.

None of the other ferrous metal alloys has a resistance to corrosion which even approaches that of the stainless steels in general, or of the austenitic nickel–chromium steels in particular. They are amazingly resistant to a very wide range of conditions of exposure and, as just mentioned, they have good resistance to high-temperature oxidation, though it does not follow that because a particular stainless alloy has exceptionally good resistance to aqueous environments it will necessarily be outstandingly good as an alloy for high-temperature service, or that the converse will be true. Nevertheless, in many instances the two properties go more or less hand in hand, though it should be pointed out that, whereas nickel in a stainless alloy may have a profound influence on its corrosion resistance at ordinary temperatures, it has very little effect on its high-temperature corrosion properties.

Chromium, of course, is the alloying element which is mainly responsible for the remarkable properties of the stainless steels. In small quantities this metal has very little effect on the corrosion behaviour of steel, but by the time about 1% has been added its influence begins to make itself felt and continues to do so in a fairly marked manner until the chromium content of the steel is about 10%. Above this figure the improvement in the corrosion resisting properties of the steel with increasing chromium content is much more marked and we come into the stainless range of alloys.

The other important element in the austenitic stainless steels is nickel. It is, of course, the presence of the nickel which renders these steels austenitic. Apart from this influence of nickel on the structure of the steel, it also has an effect on the general corrosion properties, rendering the steels more resistant to corrosion by solutions of low oxidising power.

Several other elements are added to commercial stainless steels, and many more have been investigated on an experimental scale. Some of the more important of these are discussed briefly later in the series.

From the point of view of general corrosion, since the stainless steels depend for their corrosion resistance largely, if not entirely, on passivity, the nature of the medium to which they are exposed naturally greatly influences their behaviour and this aspect of the subject will be dealt with in subsequent articles with particular reference to the more important applications of stainless steels, in for example, the chemical and food industries.

Types of Corrosive Attack

The austenitic stainless steels are by no means immune from serious corrosive attack under certain conditions: these rather specialised forms of attack will be considered. One particularly serious form of attack is intergranular penetration under certain conditions of heat treatment. Although the mechanism of this type of attack is still not fully understood, sufficient is known about the phenomenon generally for it to be possible to avoid trouble in practice if reasonable precautions are taken. Another form of attack which is not very frequently encountered in practice is stress corrosion. In this case, too, the mechanism of the attack is not known and it is clear that very much more work will be needed before the correct explanation is reached. Unfortunately, unlike the case of intergranular corrosion, we do not yet know sufficient about the stress corrosion behaviour of these steels to be able to counteract it, except by a stress-relieving anneal, a treatment which is not always practicable, particularly on a fabricated part. Fortunately, failures are comparatively few, or so it would appear from the number of instances in which it has been clearly established that the trouble was, in fact, due to stress corrosion. However, one hears of cases of failure of stainless-steel equipment from time to time, in which, for one reason or another, full invesigation of the cause was not made, and which have all the appearances of stress-corrosion failures, so that actual failures from stress corrosion may be more frequent than is generally supposed. In some instances it is by no means easy, even with a full examination of the failed part to decide whether the failure has been due to ordinary intercrystalline attack or to stress corrosion, since the latter manifests itself as an intercrystalline crack in some cases, though in others it is either wholely or partly transcrystalline. In such cases, the possibility of ordinary intercrystalline attack is, of course, ruled out and the failure is quite clearly due to stress corrosion.

Another form of corrosion which, under certain conditions, is prevalent with the stainless steels is pitting, and closely related to this, contact corrosion. The nature and incidence of these types of attack will be considered and indications given of means of preventing their occurrence, or at least reducing the risk of it.

As far as galvanic corrosion is concerned stainless steel usually acts as the cathode in any metallic couple and is therefore protected from attack. In many media the attack produced on the other metal is violent.

One detail of importance from the point of view of the practical application of the stainless steels is the condition of the surface of the steel when it goes into service. It has been shown on many occasions that stainless steel is more liable to attack if the surface is rough or contaminated with soil of various kinds than if it is clean and well polished. This applies fairly generally to several metals besides stainless steel, but its effect is very pronounced in the latter case. It must be emphasised that any surface heterogeneity has the effect of enabling corrosion to start more easily and it does not, in general, influence the rate of attack once started. Even a layer of grease or oil is sometimes sufficient to initiate the attack. Any particles of metallic or non-metallic materials which may have become embedded in the surface of the stainless steel during fabrication or handling are particularly liable to set up attack. A nitric acid dip at a moderately elevated temperature will usually remove most of the embedded material in the surface, and it is a wise precaution to give the steel such a dip before it goes into service.

Passivity

In common with other chromium alloys and chromium itself, the stainless steels possess the outstanding property of exhibiting, under a wide range of conditions, the phenomenon of passivity.

There is no really successful or comprehensive definition of passivity. Uhlig and Mears[1] have put forward two separate definitions which cover most cases. These are as follow :—

1. A metal active in the E.M.F. series, or an alloy composed of such metals, is considered passive when

1 "Corrosion Handbook" (Wiley), 1948, p. 20.

its electrochemical behaviour becomes that of an appreciably less active or noble metal.

2. A metal or alloy is passive if it substantially resists corrosion in an environment where thermodynamically there is a large free energy decrease with its passage from the metallic state to appropriate corrosion products.

Neither definition is entirely satisfactory, and even taken together they may cover cases which are not, in fact, manifestations of passivity. Passivity in metals is a subject which has received a great deal of attention from scientific investigators for many years, in fact since it was first noticed towards the end of the 18th century. Since then, when the first example which was recognised was the passivity produced on a piece of ordinary iron placed in strong nitric acid solution, there have been numerous theories put forward to explain the mechanism of the phenomenon. This is not the place to discuss these theories, but it may be remarked, in passing, that the most widely accepted theory at the present time is the general film theory. This stipulates that passivity is due to a generalised film, over the surface of the metal, which is invisible, insoluble, continuous and self-healing. In many cases such a film is considered to be an oxide, but may equally well be a chromate, phosphate or other type of film. It may also consist of adsorbed oxygen, not in chemical combination with the metal at all, but held at the surface by physical forces.

Uhlig and Wulff[2] have studied the question of passivity and have disputed the validity of the theory of the passivating effect of the oxide film. They put forward the idea of an oxygen film at the surface of the metal acting in much the same way as has been envisaged for the oxide film, in so far as it exerts a protective effect, though they claim that the mechanism is quite different, being, in effect, to prevent hydrogen from upsetting what they conceive as an electron-sharing of elements at the surface of the alloy.

Uhlig has shown in support of his theory of passivity, that if iron is plated or evaporated on to chromium, it is resistant to attack by nitric acid at the interface, a fact which he regards as showing that the chromium in intimate contact with the iron alters the reactivity of the surface atoms of the latter.

Guitton[3] regards the passivity of stainless steels as being due, primarily, to the chemical adsorption of oxygen on the surface of the metal. He showed that if the metal is sensitised by corrosion in acid prior to being passivated in contact with an oxidising agent, the passivation takes place more rapidly and at the same time is more firmly established.

Fontana and Beck[4] support the theory that a physically adsorbed gas film is the cause of passivity of stainless steels. Using a straight 18 : 8 type of steel, which they passivated by exposure first to sulphuric acid and then to air, they showed that it became active when exposed to 10% air-free sulphuric acid or to sea-water under a vacuum. On the other hand its passivity was regained on further exposure to air.

It is unfortunately not possible merely to lay down a list of solutions in which the austenitic stainless steels are passive since in most cases there is a range of concentrations and temperatures over which passivity persists and outside which the metal is active. Moreover, it frequently happens that the solution itself is not the factor which brings about the passivity, but that it is the oxygen dissolved in the solution which is responsible. It is not surprising in such a case that the manifestations of passivity are somewhat irregular. Even in the case of a solution which itself produces passivity and does not rely on the presence of dissolved oxygen, there is not usually a clear line of division between the conditions of concentration and temperature which produce passivity and those which do not. Instead there is a more or less well-defined range of transition between the passivity-producing conditions and those under which the metal is active. Furthermore, passivity is a relative term, and to say that a metal is passive under a given set of conditions does not mean that no attack at all is taking place. In fact, there are very few conditions of exposure under which it can be said that there is a complete absence of attack.

A further point about passivity is that in order for it to be maintained there must be a constant supply of the agent producing it. For example, if passivity of stainless steel is due to the presence of dissolved oxygen in a solution, then if that oxygen is used up or is removed, for example by boiling the solution, passivity will not continue, at any rate for long.

There are a number of solutions which produce a sort of borderline passivity—e.g., sulphuric and sulphurous acids at elevated temperatures. Chloride solutions are in the same category and only a slight, and often unpredictable, change in conditions is required to change them from passivating to active solutions.

The composition of the metal will also have a profound influence on the passive range under a given set of exposure conditions. The influence of increasing chromium content is referred to elsewhere, but several other elements which may be present in the austenitic steels also influence the passivity. Nickel is, of course, the most important of these, but the effect of other elements such as molybdenum, which are mentioned elsewhere is also important. The effect of carbon in contracting the passive range is discussed separately, since the manifestation of activity in this case is confined to the grain boundaries, and the corrosion which takes place is intercrystalline.

It is quite evident from these remarks on passivity that it is not possible to lay down a list of solutions with which the stainless steels can safely be used and a list with which they cannot be used. Temperature, concentration, purity, composition of the steel, all have an effect in determining the width of any passive range, and must be defined precisely. In a subsequent article it is proposed to consider a number of the more important solutions likely to be handled in stainless steel and to indicate as closely as possible the conditions under which the austenitic stainless steels can safely be used.

Galvanic Corrosion

The exact behaviour of stainless steels in a galvanic couple will depend not only on the nature of the other metal but also on whether the stainless steel itself is in the active or passive state under the conditions of exposure existing. In the passive state, of course, stainless steels are among the most cathodic of the metals normally encountered and would, therefore, not be expected to suffer any corrosion when coupled with a less noble material. This is what happens in practice, and it is the other metal of the couple which tends to

2 *Trans. A.I.M.M.E.*, 1939, **135**, 494–534.
3 *Compt. Rend.*, 1948, **226**, 805.
4 *Metal Progress*, 1947, **51**, 939.

suffer, though whether or not corrosion of this second metal is appreciably accelerated by its contact with the stainless steel is, of course, dependent on several factors besides the galvanic relationship between the two metals. Under conditions which favour the accelerated corrosion of the less noble metal of the couple, the stainless steel will tend to be protected from corrosion even more than it is already protected by its own inherent passivity, and there appears to be scope for taking advantage of this fact in protecting stainless steels from attack in media such as sea-water where its behaviour is not always ideal. Work along these lines has been carried out, and it appears that, provided the area of the other metal is adequate, the coupling of the two does in fact confer added protection on the stainless steel. Ordinary straight carbon steel is quite adequate for this purpose and it has been observed that, when relatively small areas of stainless steel have been used in conjunction with larger areas of carbon steel in sea-water, no attack whatever is suffered by the former and the acceleration of attack on the latter is not sufficiently serious to be of any practical consequence and is, in any case, usually well spread out. Stainless steel rivets, for example, give perfectly satisfactory service in steel plates.

If, on the other hand, the conditions are such that the stainless steel is in the active condition, it will, in general, behave in much the same way as ordinary straight carbon steel would under the same conditions of exposure and linked up to the same metal, its position in the electromotive series being quite close to that of the latter metal. The writer has never actually seen an example of stainless steel which has itself suffered galvanic attack, through being coupled to another, and this form of corrosion is certainly quite unusual.

Copper and Copper Alloys*

Technical Progress in 1949

By E. Voce, Ph.D., M.Sc., F.I.M.

Copper Development Association

Joining

Soft Soldering.—Soft soldering is of such universal applicability in the fabrication of articles from copper and its alloys that the publication in the form of a monograph of the researches which have been undertaken in recent years by the B.N.F.M.R.A.[73] is valuable to the copper industry.

Some tests have been reported by Cady[74] which indicate that ordinary soft soldering operations are unlikely to reduce the hardness and strength of cold-worked copper. Seams made with a soldering iron reached a maximum temperature of 300° C. and this was only endured for 10 seconds or less. The degree of work hardness of the copper sheets used in these experiments was not stated, but the results were compared with softening tests on sheets which had been cold rolled no more than 5% from the soft condition. Though these lightly rolled sheets did not soften at the temperature in question, it is almost certain that more heavily worked material would have done so appreciably. Conclusions based on such meagre evidence are dangerous.

Electric Welding.—The now well-established technique of arc welding under a shield of inert gas has been reviewed by Huff and Kugler[75] while Jones and Huff[76] have described its application to brass, copper, silicon-bronze and Monel. For the copper-base alloys direct current under a shield of helium is recommended. Gurklis[77] confirms that the shielded arc method, using either helium or argon, gives satisfactory results with silicon bronze.

A new alloy of copper with small additions of cadmium and zirconium has been developed for seam welding electrodes and similar applications.[78] After suitable cold working and heat treatment it combines a tensile strength of 32 tons/sq. in. with an electrical conductivity of from 75–80% I.A.C.S.

Other Processes.—A hydrogen-fluorine torch has been proposed for the welding of copper, nickel and steel.[79] The torch is made with concentric copper tubing, fluorine emerging from the inner and hydrogen from the outer annular tube. In welding copper the cupric fluoride which is formed melts at a lower temperature than the copper and the operation is, therefore, self-fluxing. It is claimed that by increasing the fluorine supply at the moment the copper fuses, it is possible to obtain a uniform knife-like cut. Interesting as this process sounds it is doubtful whether it could be applied to tough pitch copper without grave risk of "gassing" due to the presence of hot hydrogen.

Setapen and Marren[80] have stated that solution heat-treated beryllium copper can be brazed with silver alloys of low melting point without detriment to the subsequent precipitation hardening properties.

Corrosion

General.—The new Corrosion Handbook[81] edited by H. H. Uhlig is packed with authentic and well indexed information on all forms of corrosion. It is essentially a book of reference and, as such, fulfils its function admirably. In addition, three comprehensive summaries of existing knowledge on specific aspects of corrosion and protection have been contributed by Colegate. These deal respectively with the practical significance of galvanic corrosion,[82] dezincification[83] and the use of inhibitors in controlling metallic corrosion.[84]

Stress Corrosion Cracking.—A timely reminder of the damage which mismanagement may cause to brass through stress corrosion cracking is afforded by the failure of a bank of Admiralty tubes in an oil refinery due to mercury from a seal-pot which had been installed in the inlet system.[85]

Compared with brass, copper is relatively immune from stress corrosion cracking. It is important to note, however, that Thompson and Tracy[86] have recently

* Continued from page 101, December, 1949.

demonstrated the possibility of cracking when certain copper-rich alloys are stressed in a moist ammoniacal atmosphere. No less than 62 materials were investigated covering various coppers (with and without phosphorus, arsenic and antimony), brasses, silicon bronzes, aluminium bronzes and cupro-nickels. Though no cracking occurred in tough pitch and oxygen-free high conductivity copper, failures were found in all the other materials tested, including phosphorus deoxidised and arsenical coppers. In many cases the time to fracture dropped to a well-defined minimum with a certain definite amount of the added element and afterwards rose, showing relative immunity as the concentration of the added element was increased. Thus phosphorus contents between 0·01 and 0·6%, namely the normal commercial range, led to rapid cracking. On the other hand, arsenic was most detrimental in a narrow range of about 0·1–0·2% and commercial concentrations of 0·3 and 0·6% were less harmful. Both silicon bronzes and aluminium bronzes were similar to arsenical coppers in that the greatest tendency to cracking occurred with concentrations below those normally employed, but brasses and cupro-nickels in commercial qualities appeared to be dangerous. Even the gilding metals, which are regarded as being much less susceptible to season cracking than brasses of higher zinc content, were found to fail after relatively short exposures to the test conditions. This somewhat disquieting work is to be followed by further investigations. Meanwhile it should be emphasised that the test conditions were very much more drastic than those of normal service.

Somewhat similar in scope has been the publication by the B.N.F.M.R.A.[87] of an extensive series of experiments on the intercrystalline failure of brasses and aluminium brasses in air, ammonia, certain aqueous solutions and molten metals. The detailed conclusions and inferences are too voluminous to be given here, but the work seems to suggest that a tendency to intercrystalline cracking may be inherent in certain stressed brasses, even in the absence of a corrosive environment, and that such agents as ammonia, chloride ions, mercury and molten solder aggravate rather than originate the trouble. Ammonia was, however, found to penetrate annealed unstressed brass in an intercrystalline manner, and also to cause transcrystalline cracking, particularly in the *beta* phase, in the presence of stress.

Corrosion of Condenser Tubes.—Freeman and Tracy[88] have carried out strictly comparative tests on a number of the normal condenser tube materials such as Admiralty brass with and without arsenic, aluminium brass, aluminium bronze, phosphor bronze and cupro-nickels including those of the modern high iron, low nickel type. The marked superiority of the last named alloy in resisting the severe conditions imposed by the test was clearly demonstrated.

Bacteriological Corrosion.—Another investigation published by the B.N.F.M.R.A.[89] deals with the promotion and acceleration of metallic corrosion by micro-organisms. Most of the tests were carried out on brasses and iron-bearing cupro-nickels and it was shown that local attack can be increased by bacteria which produce by their metabolism substances such as acids and alkalis. Although insufficient to make the whole of the liquid corrosive, concentrations of corrosive agents can nevertheless be built up in the vicinity of a colony. Some organic sulphur compounds, like cystine, formed by bacteria can accelerate pitting action and sufficient experimental evidence is reported to show that the corrosive conditions of any environment cannot be assessed by physical and chemical methods alone. The biological aspects must also be taken into account.

A further contribution to this subject has appeared in America by Evans[90] who expresses the view that more corrosion of condenser tubes is due to bacteriological action than is generally supposed. He was concerned more with polluted industrial waters than with marine conditions.

Specific Corrosion Problems.—Dealing with the chlorination of drinking water, Ingleson, Sage and Wilkinson[91] conclude that cast alpha leaded brass valves fail more by the erosive action of high pressure water, especially if lead is segregated at the seating, than by corrosion, and that chlorination of the water has little effect. On the other hand, hot-pressed leaded brasses of the alpha plus beta type may suffer penetrative dezincification *via* the beta phase, and the presence of chlorine in the water increases the attack.

Many of the metals employed in boiler feedwater systems are not based on copper, but Collins[92] found that copper alloys when exposed to condensate from a vent condenser corroded about five times as fast as the same materials in hot, hard water. Tinning greatly reduced corrosion.

Kerr and Withers[93] have shown that the corrosion of solder-coated copper in the flues of gas water heaters is greatest at the dew-point temperature of the flue gases, and that the presence of sulphur trioxide has an important bearing on the rate of corrosion.

Physical Metallurgy

Recrystallisation and Grain Growth.—The American Society of Metals[94] has recently published a series of four lectures on the control of grain size in industrial metallurgy. One of these, by Burke, reviews the fundamentals of recrystallisation and grain growth, covering a bibliography of 96 references, while another, by Burghoff, describes the practical control of grain size in copper and copper alloys.

"Secondary recrystallisation," a phrase which describes an abrupt and pronounced increase in grain size at high temperatures, is discussed in relation to O.F.H.C. copper by Kronberg and Wilson.[95] They found that the large new grains had orientations related to, but not identical with the parent grains, and that the large grains were apparently nucleated at the twin boundaries of the original structure. In a somewhat similar investigation of the deformation of copper specimens of large grain size, Hibbard[95] showed that the introduction of even a single grain boundary caused marked deviation from theoretical single crystal behaviour.

Honeycombe and Boas[97] have examined the deformation and recrystallisation of 60 : 40 brass, and report that the alpha phase deforms at lower stresses and to a greater extent than the beta. Which of the two phases starts to recrystallise first on annealing depends, somewhat surprisingly, on the rate of cooling prior to deformation.

Based on the X-ray examination of filings of cold-rolled 70 : 30 brass after annealing at various temperatures up to 400° C., Averbach[98] reached conclusions in support of the view that recovery may be a process analagous to primary recrystallisation. This accords with Balicki[99] in his criticism of the findings of Cook and Richards[100] on the recrystallisation of copper.

By a carefully controlled electrolytic polishing and etching technique Schoops[101] has revealed the presence of bright bands crossing certain crystals of alpha brass after particular annealing and cold-working treatments. The ends of the bands are generally pointed and the points sometimes extend into neighbouring grains. While the phenomenon in itself is unlikely to be of much practical importance, it has enabled the author to advance interesting speculations. Two possibilities are suggested for the origin of the bands, namely the formation of mechanical twins and intense local cold working. The fact that the author has succeeded in representing these processes diagrammatically in relation to the crystal lattice is in itself a useful contribution to scientific thought.

The Fracture of Metals.—The mechanism of the fracture of metals under stress has, in recent years, received a great deal of attention, especially in America, though it formed the subject of the Institution of Metallurgists refresher course at Ashorn Hill this autumn. An important symposium was held last year by the American Society for Metals.[102] It comprises 19 papers, including contributions from such well-known authorities as Sachs, Zener, Bridgman and Hollomon, among others, and does much to clarify existing information on this fundamentally important and practical subject.

Miscellaneous

Secondary Metals.—In a report to the United Nations Economic and Social Council, Miller[103] has presented an admirable review of the supply and industrial applications of scrap metals. He estimates that as much as 65% of the copper put into service is ultimately recoverable, while about 20% or rather more of the copper supplied to the user industries is returned as scrap. These figures include alloyed copper as well as the metal itself. The total amount of copper coming forward in the form of scrap for the world in each year is said to be about 1,300,000 tons. While the paper by Miller gives no more than the briefest outline of the methods by which copper-base scrap is reabsorbed into industrial circulation, a recent publication by Whitaker[104] has presented a detailed review of the published information on this subject, including a number of flow sheets, with special reference to the recovery of metals other than copper.

Clock and Instrument Manufacture.—For hundreds of years brass has held a dominating position in the manufacture of clocks, watches and similar instruments, and it is equitable, therefore, that Tricker[105] in reviewing the metals used in the industry lays much stress on this material. Both for turned parts and for components blanked and formed from strip-leaded types of brass are used, and it is interesting to note that a British Standard for clock brass is at present in preparation. Nickel silver, usually leaded, is employed in some cases to replace brass, mainly for the sake of appearance, while phosphor bronze, cadmium copper and beryllium copper are preferred to steels for hair springs. Turning from clocks to instruments, 80 : 20 gilding metal is a favourite material for bellows, while pressure sensitive capsules and Bourdon tubes are frequently made of beryllium copper. Phosphor bronze is also employed for the last named purpose, while oil impregnated porous bronzes are finding increasing application, especially for the fast moving gear trains.

The Oldest Known Metal Tools.—It is perhaps trite to remark that copper was the first base metal to be employed by man, and that it was followed at a comparatively early date by bronze. Cairo Museum is soon to display a magnificent collection of tools from a tomb believed to be that of King Zer who reigned about 3,000 B.C.[106] The hoard includes copper axe blades, knives, engraving tools, piercers, bodkins, needles, saws, chisels, adzes and curious copper plates of unknown purpose. It is believed that copper saws were used to cut stone as well as wood, and the slow and laborious nature of the process can well be imagined.

A New Compendium.—In conclusion a welcome must be extended to the comprehensive collection of metallurgical data which has been compiled under the editorship of Smithells.[107] It is an indispensable addition to any metallurgical library.

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Automatic Control of Electrodes in Arc Furnaces

By G. Reginald Bashforth, F.I.M.

The automatic electrode control gear is an important part of the electric arc furnace ; its function is to ensure, by adjustment of the electrodes, a near constant power input for a given setting of the power controls. Two types of regulator are discussed and reference is made to applications.

IN a recent article[1] on electric furnaces in the metallurgical industry, the automatic control of electrodes was briefly mentioned. The brevity in which this subject was treated was due to the fact that the matter was regarded as an electrical engineering problem rather than one which concerned the metallurgist. The automatic electrode control gear, however, is a very important part of the arc furnace ; its function is to ensure by adjustment of the electrodes that the power input remains practically constant for a given setting of the power controls. Good control simplifies attendance and improves the economic operation of the furnace.

As mentioned in the previous article, for a given current the heat input depends on the length of the arc formed between the electrodes and the bath of metal. The heat or power input must also be regulated to meet the conditions or stages of the process. The heat liberated in the furnace, that is, the power input, must be fixed and controlled according to the ability of the charge to absorb that heat. As shown by Lewis[2] if the charge is unable to absorb this heat any surplus energy will be absorbed elsewhere within the furnace and will react unfavourably on :—(1) The furnace lining ; (2) the carbon pick-up ; (3) the electrode consumption ; and (4) the power consumption.

During the meltdown the power input will be set at a higher value and the load will fluctuate violently and rapidly between comparatively wide limits. Later in the process, when a semi-liquid bath of metal exists, a lower power input is required and the fluctuations are less violent. In the final stage or stages, when the charge is completely melted, still less power is required and the load should be substantially stable and constant. Any method of electrode control or positioning must be able to meet these requirements.

McBroom[3] shows that any system of electrode control consists of two parts :—

1. The electrical component, by which the impulses from the current and/or voltage fluctuations are relayed, amplified and caused to activate suitable positioning gear.
2. The mechanical component by which the electrodes are actually raised or lowered.

He suggests that the dividing line between these two parts can be drawn at the output side of the motor which positions the electrode arm.

If a short circuit occurs, for example due to a piece of scrap falling against an electrode in such a manner as to contact the electrode and the metallic bath at the same time, a surge of current results. The resulting increase of current in the electrode circuit causes a corresponding increase of current in the secondary of the electrode current-transformers. This increase of current is made to serve, through suitable relays and amplifiers, the motor for positioning the electrode. The power input into this motor is in a direction which will cause the electrode to lift away from the bath.

Conversely a reduction in the electrode current, due to the falling away of the scrap or any other reason, would have the opposite effect. The resultant power input into the motor would be in the opposite direction so as to cause the electrode to be lowered towards the bath.

In the article, to which reference has already been made, the two components of the electrode control device were shown in Figs. 5 and 6. The actual principles, however, shown in Fig. 5 were not these employed by Birlec, Ltd. in connection with the mechanical application of electrode control used on their furnaces and reproduced with their permission in Fig. 6.

Stansel[4] discusses two types of regulator—the intermittent and the continuous, both of which are automatic. Each phase is regulated independently and the three elements of a three-phase regulator combine to maintain a balanced load on the power system, that is, so that equal current is maintained in each of the three phases from the main power supply.

The amount of current in each electrode can be adjusted by several means, but generally by a rheostat

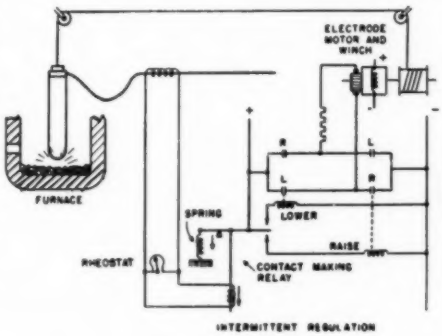


Fig. 1.—The principles of the intermittent type of regulator.

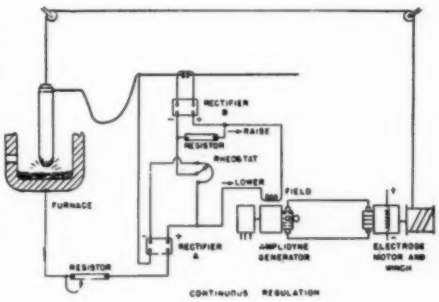


Fig. 2.—The principles of the continuous type of regulator.

1 Bashforth, *Metallurgia*, Sept., 1949.
2 Lewis, J., *Iron and Steel*, May, 1946.
3 McBroom, J., Electric Furnace Steel Conf. A.I.M.E., 1943.
4 Stansel, N. R., Electric Furnace Steel Conf. A.I.M.E., 1943 ,

suitably connected in the circuit. The term " rheostat " is employed in a wide sense and includes adjustable potentiometers.

With the *intermittent type* of regulator shown diagrammatically in Fig. 1, any increase or decrease of current in the furnace circuit outside the limits for which the contact-making relay is set closes the control circuit of the electrode motors. This action would result in the raising or lowering of the electrodes as required. There is, thereby, an intermittent correction of the length of the arc so as to maintain an average value of the current in the furnace circuit.

The general principles of the *continuous type* of regulator are shown diagrammatically in Fig. 2. These principles consist of opposing the voltage of a current transformer in the furnace circuit by a constant or reference voltage. The reference voltage is obtained from the rectifier A whilst the opposing voltage is derived from the rectifier B. The voltage of the rectifier A, which is a function of the voltage from the electrode to the furnace shell, tends to send the current through the field of the " Amplidyne " generator so as to lower the electrode. The voltage from the rectifier B is determined by the current in the furnace circuit. This voltage from B tends to send the current in the opposite direction to the current from A, thereby causing the electrodes to rise. The movement of the electrodes is, therefore, determined by the relative value of the voltages from A and B.

Any increase in the current in the arc due to a reduction in the length of the arc, results in a corresponding decrease in the voltage across the arc. When the electrodes are lowered into the metal the maximum current is passed. This increase of current in the furnace circuit increases the voltage from the rectifier B, thereby raising the electrodes. The increasing distance between the electrode and the bath results in an increasing voltage between the electrode and the furnace shell, whereby the voltage from the rectifier A is increased.

This increase in the voltage from the rectifier A, tends to lower the electrodes. This opposition is continuous whilst the furnace is in operation, resulting in the continuous maintenance of the length of the arc corresponding to the current in the furnace circuit for which the regulator is set.

This second method of control takes cognisance of both the voltage and current. Since the resistance of an electric arc varies with the length of the arc, the longer the arc the higher the voltage across it and the smaller the current through it, or vice-versa. This is an important feature of the continuous type of regulator, which is applied to most modern furnaces.

As pointed out by Lewis,[5] the " Amplidyne " control gives a continuous control of the electrodes, relying not on impulses, but on the continuously varying degree of out of balance from the present voltage/current relationships. The power developed in the electrode control system is directly proportional to the degree of out of balance at any time, and the speed of correction is thus smoothly varied to give no more and no less than the movement required.

According to a recent publication[6] two sensitive types of electrode control are tending to replace the balance beam type in America. The Regulex (by Allis Chalmers) has separate direct current generators to operate the motor positioning each electrode. These generators are fed back with a neutralising or reversing current, according to the EMF developed across the arc.

The Westinghouse Electric Manufacturing Company has an entirely static system, employing electronic amplification to feed back in order to supply sufficient power to operate the motors controlling the electrodes. This electrical apparatus can be made to activate suitable mechanical, hydraulic or other convenient means to position the electrodes. This mechanism varies on different types of furnace. A neat application employed on the " Birlec " furnace is shown in Fig. 3.

The actual application of these principles of control varies with the different makes of electric furnaces, and it is beyond the scope of this article to discuss the merits and demerits of any particular type. Sometimes the metallurgist dealing with electric furnaces is handicapped by a limited knowledge of electrical terminology and engineering. Recently two very helpful and interesting papers dealing with the fundamentals of electricity for the melter were presented by Oltrogge and Cramer.[7]

The Author would like to express his thanks to Mr. R. Paterson, Electrical Engineer at the Round Oak Steel Works, Ltd., formerly of Messrs. Stewart & Lloyd, Ltd., Corby, for his help and criticism in the preparation of this article.

Courtesy of Birlec Ltd.

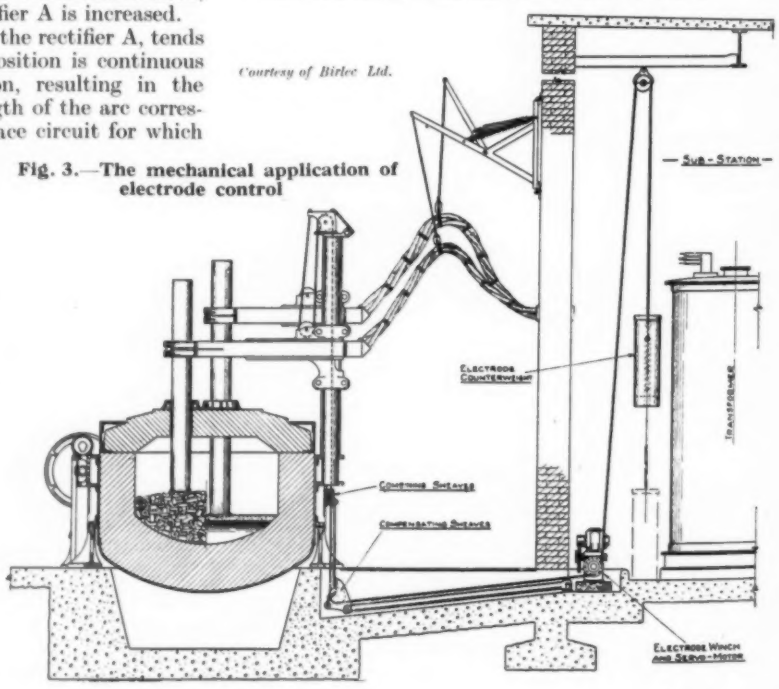


Fig. 3.—The mechanical application of electrode control

5 Lewis, F. V., Private Communication from Sales Metallurgist Birlec.

6 Steel Founding, Anglo American Council on Productivity, Sept., 1949. 21 Tothill St., S.W.1. Price, 3s.

7 Oltrogge and Cramer, Electric Furnace Steel Conf. A.I.M.E., 1947.

The Effect of Homogenising on the Fatigue Properties of an Extruded Al—Cu—Mg Alloy

By H. A. Unckel and H. H. Nyander

Due to the presence of streaks of inclusions of intermetallic compounds, extruded alloys of the Duralumin type exhibit inferior mechanical properties in the transverse direction, as compared with those in the longitudinal direction. In the November issue, one of the authors reported the results of an investigation into the effect of homogenising treatments on the static tensile properties and the present article concerns an extension of that work to the dynamic properties. Some considerations of the mechanism of fatigue are also presented.

IN a previous article[1] the effect of homogenising treatments on the static properties—yield stress, ultimate tensile stress and elongation—of extruded rods of an aluminium alloy containing 4·6% Cu, 1·35% Mg, 0·59% Mn, 0·25% Si, and 0·42% Fe was discussed. The present article is concerned with fatigue tests on the same material and, as the time of homogenising appeared to have no appreciable effect on the mechanical properties, the fatigue test results quoted are confined to longitudinal and transverse tests of two batches of specimens. Both batches had been solution-treated and aged but, in one case, the material was homogenised for 15 hours in the extruded condition.

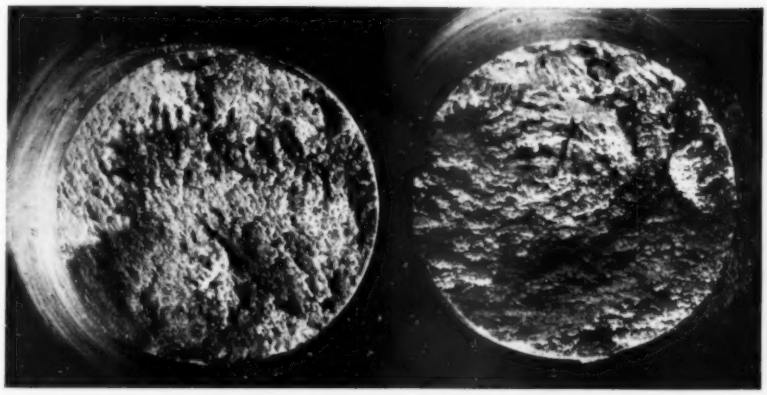


Fig. 1.—Appearance of fatigue fractures at a magnification of 5. Left: Longitudinal test-piece. Right: Transverse test-piece. The fractures started at the upper periphery and in the case of the transverse test-piece the direction of the inclusion streaks is from left to right.

Type of Test Used

Fatigue tests were carried out on a machine of the rotating-beam cantilever type. The total length of the test-piece was 100 mm. and the diameter of the head was 15 mm. For a length of 75 mm. the diameter was turned down to 10 mm., with a 5 mm. radius at the junction of the two diameters. The lever arm, i.e., the distance between the point of loading and the fillet was 60 mm. After turning, the surface of each specimen was finished with a fine abrasive paper. The maximum stress is given by the expression

$$\sigma = \frac{32Pl}{d^3}$$

where P is the applied load.
l is the lever arm.
and d is the diameter.

In the case of steel, it is only necessary to run fatigue tests to about 10 million reversals of stress in order to determine the fatigue limit. With aluminium and its alloys, however, it is known that a definite fatigue limit does not exist, i.e., the load which can be supported without fracture decreases as the number of reversals is increased. It is, therefore, customary to run tests for more than 100 million reversals—in some cases tests have proceeded as far as 500 million reversals. In the present investigation it was considered that 25 million reversals would suffice for the differentiation between homogenised and unhomogenised material and between tests taken in the transverse and longitudinal directions. To avoid the "training-up" effect, each test piece was run at one load only, the test being discontinued after 25 million cycles. On the basis of a previous investigation[2] on the fatigue properties of extruded light alloy rod of 50 mm. diameter, the fatigue strength was expected to range from ±11 to ±15 kg./mm.² and loads within these limits were accordingly chosen.

Results Obtained

The results obtained are set out in Table I, the differently treated test material being grouped, for ease of comparison, according to the load applied. These values were plotted in the usual manner, viz., stress versus the logarithm of the number of reversals.

As far as can be concluded from a relatively small number of tests, there is no significant difference between the results for the two batches of material—homogenised and unhomogenised. The longitudinal test results show a slight superiority over those for the transverse test-pieces, although the difference is nearly overshadowed by the scatter, which is appreciable. As mean values for the limiting safe stress at 25 million reversals, ±14 kg./mm.² for the longitudinal and ±12·5 kg./mm.² for the transverse direction may be adopted. This difference is considerably less than the difference in

1 Effect of Homogenising on the Structure and Properties of an Extruded Al-Cu-Mg Alloy. H. A. Unckel, METALLURGIA, 1949, **41**, 241, 26–29.

TABLE I.

Spec. Load kg./mm.²	Treatment	Specimen	Number of reversals (millions)					
15·3	Not homogenised	Longitudinal	2·5	3·1				
,,	,,	Transverse	0·5	1·8				
,,	Homogenised	Longitudinal	1·0	2·5	4·9			
,,	,,	Transverse	1·0					
14·7	Not homogenised	Longitudinal	2·6	3·2	22·6	>25		
,,	,,	Transverse	0·6	0·9	5·2	5·6		
,,	Homogenised	Longitudinal	13·8					
,,	,,	Transverse	1·1	2·2				
14·1	Not homogenised	Longitudinal	6·8	>25	>25			
,,	,,	Transverse	3·0	13·3	>25			
,,	Homogenised	Longitudinal	0·7	4·7	>25			
,,	,,	Transverse	0·8	2·0	3·0	4·9	5·0	5·6
12·9	Not homogenised	Longitudinal	>25					
,,	,,	Transverse	3·0	13·3	>25			
,,	Homogenised	Longitudinal	7·3	>25				
,,	,,	Transverse	25					

the static tensile stresses, which amounted to as much as 20–25%.

Brenner and Kostron[3], in an investigation into the fatigue properties of extruded duralumin bars with a cross-section of 50×90 mm., using a flat bending machine, found values of 15 kg./mm.² for the longitudinal tests and 12·5 kg./mm.² for test-pieces taken at 45° to the longitudinal direction.

Effect of Inclusion Streaks

Fatigue cracks always start at the surface of the test-piece, since the stress is at its maximum there. On the assumption that cracks tend to develop preferentially at inclusions, due to a disturbance of the stress distribution, some difference in the fatigue values in the longitudinal and transverse directions would be expected. In the cross-section of the extruded bar the inclusions are evenly distributed and isolated from each other by the homogeneous matrix. On the other hand, in the longitudinal section, i.e., in the cross-section of the transverse test-pieces, the streaks of inclusions provide localities of easy crack initiation and paths of more rapid propagation.

In this connection, it is interesting to compare the longitudinal and transverse fatigue strengths of wrought iron, in which the inhomogeneity consists of streaks of slag particles instead of intermetallic compounds as in the present case. The Metals Handbook (American Society of Metals) gives values, for wrought iron, of 23,000 p.s.i. (16·2 kg./mm.²) in the longitudinal direction and 19,000 p.s.i. (13·3 kg./mm.²) in the transverse direction.

In the rotating-beam method of fatigue testing, the fracture starts from one or more points on the periphery and proceeds steadily inwards until the remaining section is no longer able to support the load, when failure occurs suddenly as in a static test. Examination of the fractured surfaces showed that, with both the longitudinal and transverse test-pieces, the final fracture was half-moon shaped (convex towards the centre of the section) and lay at the periphery. This indicates that the first fatigue cracks originated from some point (or series of adjacent points) on the opposite section of the circumference. In work reported previously[2] it was found that, with alloys of the type Al–Cu–Mg (but not with those of the type Al–Mg–Si), the fatigue cracks on the transverse test-pieces had started from that part of the periphery which is at right angles to the direction of the inclusion streaks, i.e., where the streaks cut the surface tangentially. The same effect was observed in the present work, with few exceptions. There seems to be a certain amount of justification, therefore, for the conclusion that the streak-like arrangement of the intermediate phases plays some part in determining the fatigue characteristics, although the preferred orientation in extruded bars may also be a factor.

Absence of Fatigue Limit

Although not pertaining to the purpose of the present investigation, some observations on the absence of a definite fatigue limit in the case of aluminium and its alloys may not be out of place. This property seems to be exhibited by all metals with relatively low melting points. Gough, in his classical work on fatigue[4] attributes the initiation of a fatigue crack to the effect of crystallographic slip, a view which is confirmed by almost all the existing evidence, and the mechanism of fatigue may be imagined to be of the following nature. Even if the nominal stress is below the static yield stress, there will always exist points in the structure at which the local stress is higher, as a consequence of uneven stress distribution due to such irregularities as voids, regions of lattice imperfection at grain boundaries or mosaic-block boundaries, or grain fragmentation caused by plastic slip. If the surroundings of such weak spots are sufficiently strengthened by strain-hardening in the course of repeated straining, spread of a crack may be prevented and the load supported indefinitely. On the other hand, if the strain-hardening effect is insufficient, the crack spreads until failure occurs. Metals and alloys with a low temperature of recovery from strain-hardening (i.e., with a low melting point) cannot, therefore, be expected to have an appreciable fatigue limit. With very pure aluminium, recovery from cold work takes place at or near room temperature, but the recovery temperature is increased considerably by impurities or alloying elements which also raise the yield stress. Furthermore, it is well known that the recovery and recrystallisation temperatures are lower, the greater the amount of cold work. By repeated local deformation, the temperature of the strained region is raised and strain-hardening under fatigue conditions cannot, therefore, exceed a certain amount. With age-hardened Al–Cu–Mg alloys, the high static strength is a consequence of the lattice distortion accompanying precipitation hardening. Plastic deformation, as is well known, tends to accelerate the actual precipitation of the solute atoms, thereby reducing the lattice distortion and, therefore, the hardening effect. Thus plastic deformation in itself, together with the local temperature rise resulting from it, limits the effect of strain-hardening in preventing the spread of fatigue cracks and the longer the fatigue test is run, the lower will be the load supported.

Change of Address

We are informed by Messrs. Thomas Andrews & Co., Ltd., steel manufacturers, Sheffield, that the rebuilding of their offices at Royd's Works is now completed and the temporary offices at 8, Palmerston Road, Sheffield are closed. All communications and goods intended for Palmerston Road should be addressed or delivered to Royd's Works, Attercliffe Road, Sheffield (P.O. Box 68). Telephone No. Sheffield 22131.

2 Fatigue tests with extruded bars of light alloys in the longitudinal and transverse directions. H. A. Unckel, *Metallwirtschaft*, 1942, **21**, 427–429.
3 P. Brenner and H. Kostron. *Luftweissen*, 1938, **51**, 15–16.
4 Edgar Marburg Lecture. H. J. Gough, *Am. Soc. Test. Mat.* 1933, 33.

Some Factors Influencing the Use of Ethyl Silicate in Precision Casting

By H. G. Emblem, A. E. Meadowcroft, and G. E. Stockwell

Research and Development Department, Monsanto Chemicals, Ltd.

Results are given of an investigation carried out to evaluate the factors governing the suitability of a given sample of commercial ethyl silicate for use in lost wax precision-casting processes.

DURING the past twelve months, the quantity of ethyl silicate used by precision-casting foundries in this country has increased very considerably. Much of this increase is due to the growing production of castings other than those connected with the gas-turbine engine. Accordingly, therefore, an investigation was planned to evaluate the factors governing the suitability of a given sample of commercial ethyl silicate,* for use in lost wax precision-casting processes.

Precision Casting—Use of Ethyl Silicate

A number of descriptions of the lost wax precision-casting processes have been published in the last few years.[1, 2, 3, 4] Recently, Short has described a typical commercial operation of the process.[5] Ethyl silicate is used as a binder in these processes because :—

(i) Extremely accurate castings can be obtained.
(ii) No alkali metals (sodium, potassium) are present ; this minimises slagging troubles.
(iii) Moulds, called investments, resulting from its use are highly refractory.

Consequently, intricately shaped components, of close, reproducible dimensional tolerances and good definition, may be cast in high melting-point alloys, using ethyl silicate bonded investments and spray or dip coatings.

Before ethyl silicate can function as a binder, it must first be reacted with water (hydrolysed). This reaction is speeded up (catalysed) by the addition of any strong acid, or alkali. Regardless of the catalyst used, a gel is formed akin to those which can be obtained from sodium silicate. These gels are complex silicic acids built up from the fundamental unit orthosilicic acid, $Si(OH)_4$.

During the process of setting, water and ethyl alcohol are liberated. The setting sequence takes place in several stages, which may be represented as :—

(i) Hydrolysis—to give silicic acid, and ethyl alcohol.
(ii) Condensation—to give complex silicic acids.
(iii) Further condensation—to give a coherent gel structure.
(iv) On firing, the gel forms an intercrystalline compound with the refractory investment. This has been experimentally demonstrated in the case of alumina fillers, some mullite formation occurring.

Characteristics of the Action of Ethyl Silicate

A number of factors control the reproducibility of the gelation process. It is proposed to discuss only those of direct importance in lost wax precision-casting operations. The most important variable is the nature of the catalyst for hydrolysis and gelation.

Ethyl silicate and water are not normally miscible but become homogeneous when a mutual solvent such as ethyl alcohol is added. During hydrolysis of ethyl silicate, extra ethyl alcohol is produced. It, therefore, follows that a mixture of ethyl silicate, water and insufficient alcohol to give complete initial miscibility, will tend to become homogeneous as hydrolysis proceeds. Consequently by suitable adjustment of proportions the mixture may be made to become homogeneous at the end of hydrolysis, thus giving a visible indication of the completion of the reaction.

An alternative indication of the progress of the hydrolysis reaction is obtained by observing the temperature of the mixture. The temperature at first rises slowly, then rapidly to a peak. This peak temperature denotes the completion of hydrolysis. If the reactant proportions are such that the system is initially homogeneous this peak temperature of hydrolysis method is the best way to ascertain completion of reaction.

Hydrolysis and Gelation of Ethyl Silicate

The effect of an acid catalyst was first studied. Hydrolysis times varying from less than 1 minute to 24 hours were obtained experimentally. These variations were brought about entirely by changes in the concentration of the acid catalyst, in this case, hydrochloric acid. Prior work on ethyl silicate hydrolysis using hydrochloric acid as catalyst has been summarised by Cogan and Setterstrom.[6]

In order to assess the influence of the acid catalyst on the hydrolysis and gelation sequence, the proportions of ethyl silicate, water and ethyl alcohol were fixed at : 100 grms. ethyl silicate, 48 grms. (60 ml.) absolute ethyl alcohol ; and 17·5 grms. water. A number of such mixtures were prepared in stoppered bottles, known concentrations of hydrochloric acid being present in the water. The times required for the mixtures to gel were observed.

All the mixtures became homogeneous on standing, and gelled after a further time interval. The shortest gelling time recorded was 18 hours, the longest 215 hours. Table I gives some typical results. The experiments demonstrate the wide range of hydrolysis conditions which may be employed.

* The ethyl silicate used throughout this work was the commercial product made from silicon tetrachloride and 64 O.P. industrial methylated spirit and containing approximately 40% of silica.

1 Dunlop, *Foundry Trade J.*, 1945, **75**, 108.
2 Glaser, *Iron Age*, 1945, **157**, 52.
3 Read, *Metallurgia*, 1947, **35**, 275.
4 Evans, Sutton and Thexton, *Metal Ind.* (London), 1947, **71**, 3–6 ; 23–6. See also *Chem. Abs.*, 1947, **41**, 5840 b.
5 Short, *Aust. Production Engineers*, 1948, **27**, No. 2 (Feb.). See also *Aircraft Engineering*, 1948, **20**, No. 230, 105.

6 Cogan and Setterstrom. *Ind. Eng. Chem.*, 1947, **67**, 1364.

TABLE I.—GELATION TIMES OF FULLY HYDROLYSED ETHYL SILICATE SOLUTIONS

Experiment Identity	Residual HCl in Ethyl Silicate	Amt. HCl from Aqueous Solution	Water Content of Mixture	Gelation Time
1	0·076 grms.	22 ml. conc. HCl	17·5 grms.	18·0 hrs.
2	" "	4·588 grms.	"	29·5 "
3	" "	4·588 "	"	28·5 "
4	" "	2·147 "	"	66·0 "
5	" "	2·147 "	"	67·0 "
6	" "	1·033 "	"	140·0 "
7	" "	1·033 "	"	132·0 "
8	" "	0·518 "	"	213·0 "
9	" "	0·518 "	"	215·0 "

With the exception of experiment No. 1 the hydrochloric acid was added from specially prepared aqueous solutions. In experiment 1, commercial concentrated hydrochloric acid was used.

Other proportions of ethyl silicate, ethyl alcohol and water may, of course, be used. A variation in these proportions, will obviously influence the time required for hydrolysis and gelation. Typical formulations of interest in general foundry practice, with details of their use, have been given by Tedds.[7]

Gelation may also be accelerated by warming the hydrolysed solution. This has been used as a method of setting investment mixtures. Normal climatic fluctuations, however, are not sufficient to cause critical time differences in the gelation of a particular hydrolysed ethyl silicate solution. In practice, the use of strongly acid solutions is not advisable, because of manipulation difficulties. With low acid concentrations, the times of gelation at normal temperatures are often inconveniently long for works usage.

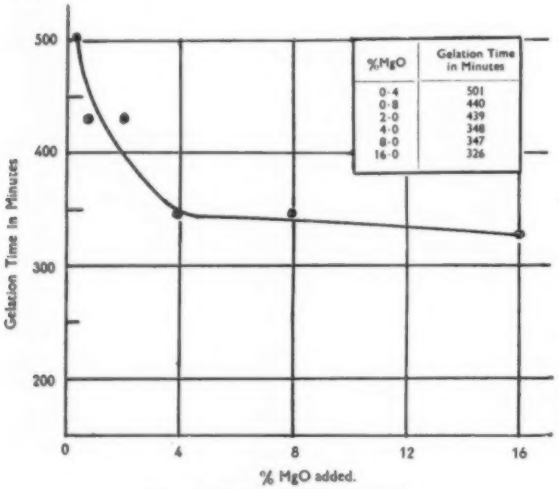


Fig. 1. Gelation time graph.

Gelation time of hydrolysed ethyl silicate solutions at normal atmospheric temperatures may, however, be greatly decreased, by the addition of any alkali. The refractory mould materials normally used are of themselves usually insufficiently alkaline to cause any significant shortening of the gelling time. It is, therefore, necessary to add extra alkali to the mould material, before the latter is mixed with the bonding solution. The alkaline agent most commonly used is light calcined magnesium oxide, which has the advantage of being highly refractory, and does not break down to give gaseous by-products.

7 Tedds, *Foundry Trade J.*, 1949, **87**, 286, 315.

Effect of Gelation Accelerators

In order to assess the influence of the amount of magnesia on the gelling time of a hydrolysed ethyl silicate solution, varying amounts of calcined light magnesia were added to samples of a standard hydrolysed ethyl silicate solution. Mixing was carried out by shaking in a stoppered bottle, the viscosity of the mixture being observed at intervals. When the whole mass fell from the sides and bottom of the inverted bottle, it was considered to be set. The time taken to reach this state from the initial time of mixing was recorded as the setting time. Fig. 1 shows the results obtained, and indicates that under these conditions a minimum amount of 2% w/v magnesia is required to obtain reasonably rapid setting times. As magnesia is insoluble in the solution, reduction in setting times resulting from amounts greater than this are considered to be due to the increase of alkaline surface. This effect is greatly accentuated when the magnesia, instead of being dispersed throughout the solution, is distributed through the filler, which may of itself be alkaline. Standardisation of the filler alkalinity, beyond determining that it is above a minimum figure is therefore unnecessary.

Control Analysis of Ethyl Silicate for Precision Casting

Assessment of the suitability of a given sample of commercial ethyl silicate for use as a binder in lost wax precision-casting processes will therefore require :—

(i) Determination of the free acid present in the sample, since this will affect the stability of the hydrolysed solution prepared under standardised conditions.

(ii) Determination of the time required for this hydrolysed solution to set when mixed with a sample of a standard refractory mould or investment material, or " filler," and the determination of the alkalinity of this filler. The refractory filler should be representative of those generally used in commercial practice.

Methods for carrying out these determinations have been developed, and are reproduced below.

(i) Determination of Free Acid in Ethyl Silicate

Method.—Hydrochloric acid is estimated by direct titration of an ethyl silicate—water suspension with standard alkali, using a methyl orange indicator. The end point is ascertained by comparison with reference solutions in which the indicator shows the acid and alkaline colours respectively.

Reagents.—Saturated solution of methyl orange in ethyl alcohol. Standard N/10 alkali.

Procedure.—First prepare the reference solutions. The acid reference solution is prepared by shaking 50 ml. ethyl silicate, 50 ml. distilled water, and 1·0 ml. methyl orange indicator solution for 15 minutes in a 250-ml. stoppered conical flask. The alkaline reference solution is prepared by shaking 50 ml. ethyl silicate ; 50 ml. N/10 alkali and 1·0 ml. methyl orange indicator solution for 15 minutes in a 250-ml. stoppered conical flask.

Weigh exactly 50 gm. of ethyl silicate into a 250-ml. conical flask. Add 50 ml. distilled water, and 1·0 ml. methyl orange indicator solution. Close the flask, shake for 15 minutes, then titrate immediately with standard decinormal alkali. Shake at frequent intervals during the titration. The end point is when the solution shows

an orange tint, midway between the acid and alkaline colours. Report the result as % HCl w/w.

$$\%HCl\ (w/w) = (\text{vol. of alkali}) \times \text{Normality} \times 0.073$$

(ii) Hydrolysis and Setting Test for Ethyl Silicate

Method.—Ethyl silicate is hydrolysed using an acid catalyst, and the ability of the hydrolysed solution to bind a refractory filler, is assessed. The alkalinity of the filler is also determined.

Reagents.—Standard N/1 acid solution. Standard N/1 alkali solution. Hydrochloric acid solution, 0·06% w/v (pH 1·8). Refractory fillers, of specified sieve grading. Phenolphthalein indicator solution (approx. 1% in ethyl alcohol). 64 O.P. I.M.S. (Industrial Methylated Spirit).

Procedure.—The hydrolysed ethyl silicate solution is prepared as follows :—

Sixteen ml. of 64 O.P. I.M.S. and 12 ml. of hydrochloric acid solution [0·06% w/v (pH 1·8)], are mixed and 76 ml. of ethyl silicate then added. The whole is shaken in a wide-mouthed stoppered bottle until the mixture clarifies and the temperature rises. Note that this temperature rise may be quite small, approximately 5°–10° C. One hour after clarification add a further 60 ml. of ethyl silicate. When the solution has cooled to approximately room temperature, it is ready for use.

This solution is used to bind a filler comprising :—

Fire clay grog	72·5% w/w
Charp..	22·5% w/w
Gelation Accelerator	5·0% w/w

The gelation accelerator mixture comprises 5·0 gms. of light magnesia and 95 gms. of charp. The filler composition employed may be compared with that quoted by Short.[5] It is most important that mixing in all cases should be thorough and uniform to ensure even dispersion of the magnesia. Failure to observe this precaution leads to unreliable results.

Eighty-five grm. of the above filler are mixed with 25 ml. of the hydrolysed ethyl silicate solution. This mixture is poured into a glass mould greased with vaseline. The mould is a hollow glass cylinder 2 in. diameter × 1½ in. high, attached to a porcelain tile by an external ring of plasticine. Observe the time required for the mixture to set to such an extent that it does not sag under gravity when the mould is removed. Report this time as the setting time. An indication of setting can be obtained by pressing gently on the surface of the mix with the finger before removing the mould.

The filler alkalinity is estimated as follows :—

Twenty-five gms. of filler are accurately weighed, and digested with 25 ml. of standard N/1 acid solution for at least 15 minutes. After digestion, filter the residue, and wash thoroughly with distilled water. Collect and bulk all washings and filtrates. Titrate the excess acid with a standard N/1 alkali using phenolphthalein as indicator. Report the alkalinity as percentage MgO.

$$\%\ MgO = \left[25 - \frac{(\text{Vol. of alkali})\ (\text{Normality of alkali})}{(\text{Normality of acid})}\right] \times 0{\cdot}08 \times (\text{normality of acid})$$

Tolerances

To obtain the best results in lost wax precision casting a sample of ethyl silicate when analysed by the methods outlined above, should have an acidity, as hydrochloric acid of the order of 0·07% (w/w), and a setting time from 1 to 3 hours. The upper limiting figures are an acidity as hydrochloric acid, of 0·10% (w/w) and a setting time of 3 hours. Much lower figures than these are desirable. Table II gives results for some typical commercial ethyl silicate samples.

TABLE II.—ANALYTICAL RESULTS FOR SOME TYPICAL SAMPLES OF COMMERCIAL ETHYL SILICATE

Sample	Acidity as Hydrochloric Acid	Setting time (hours)	Remarks
1	0·075% (w/w)	1½	Suitable for use in lost wax precision-casting process.
2	0·03% (w/w)	1	
3	0·10% (w/w)	3½	Unsuitable, long setting time.
4	0·12% (w/w)	2½	Unsuitable, high acidity.

It is hoped that the above information will serve a twofold purpose. Firstly, that it will confirm the experience of established users of ethyl silicate, and secondly, be of assistance to foundries considering production by investment casting processes, or other uses for ethyl silicate.

Production Efficiency Research in the Machine Shop

A STRIKING example of the value of science to industry was provided recently by an investigation completed by the Production Engineering Research Association. The cost of the work was less than half of what was saved annually by the application of the results.

A firm in the Midlands was facing high costs for the cutting fluids used in their gear-cutting shop. The problem was turned over to a team of investigators from the Research Association. After researches in the machine shops of the firm and in the laboratories they discovered that the cutting fluids used were not well suited to the job and recommended others more efficient for the particular operations involved. The cost of this research was £400. The cost of the cutting fluids used immediately fell from approximately £1,500 to £500 per year and the firm was able to produce equally good or even better finished components at the same production rates as before.

Besides work on individual problems PERA carries out research on others of fundamental importance, for example on tool grinding. The methods of tool grinding used in many British workshops are wasteful and inefficient, because each worker is allowed to grind his own. Every single person regrinding a drill or tap has a different way of doing it, the grinding wheels may be worn, and so on. Consequently no drill ground will ever be identical with another, much less ground properly, unless a special drill grinding machine is used. The way to ensure that drills are ground for the maximum efficiency is to centralise the grinding, for even 5 thousandths of an inch difference in the lip height of a drill can reduce its life by half. When a tool becomes worn it should be exchanged for another, ground with the right equipment at a central point. If this were done, tools would not need to be ground nearly so often, apart from the advantage of higher speeds and more accurate work. PERA is to hold a conference on tool grinding early in 1950, and demonstrations of good tool-grinding practice will be included.

The field of PERA's work is wide. Its services to members cover the design and use of cutting tools and machine tools, forging, extrusion, die-casting, mechanical handling, surface finishing and many other processes. Firms have been helped with problems ranging from the jigging and machining of phosphor-bronze machine bodies to ink printing on metals, from costing to colour schemes for machinery.

Staff Changes and Appointments

MR. C. S. GILL, Managing Director of the Davy and United Roll Foundry, and Chairman of the Steel Roll Makers' Joint Council, has been re-elected Chairman of the Roll Makers' Association of Great Britain for the fourth successive year. He has also been re-elected Chairman of the Back-up Roll Makers' Association Chairman of the North East Coast Association of Steelfounders, and Employer Representative on the Teesside District Advisory Committee of the North Regional Board for Industry.

DR. W. A. NAISH, who relinquished his position as Head of the Metallurgy Department of the Battersea Polytechnic at the end of the Michaelmas term, has taken up an appointment as Technical Director with the Hoyt Metal Company, London. Dr. Naish is succeeded at Battersea by MR. L. W. DERRY, formerly Senior Lecturer in the department.

MR. FRANK W. ROWE, Managing Director of K & L Steelfounders and Engineers, has succeeded Mr. F. A. Martin as Chairman of the B.S.F.A.

DR. F. D. RICHARDSON will relinquish his present position as Head of the Chemistry Department of the British Iron and Steel Research Association at the end of this month. He has been appointed Nuffield Research Fellow in Extraction Metallurgy at the Royal School of Mines, London.

MR. GERALD STEEL, Joint Managing Director with the Chairman, SIR WALTER BENTON JONES, of United Steel, has been appointed sole Managing Director. SIR WALTER BENTON JONES has relinquished the Joint Managing Directorship, but still remains Chairman. MR. A. J. PEECH, Assistant Managing Director, becomes Deputy Managing Director; MR. K. G. LAMPSON, General Sales Manager, becomes Director in charge of sales; and MR. H. M. HENDERSON, Commercial Manager of Appleby-Frodingham, becomes General Sales Manager of United Steel. MR. HENDERSON is succeeded by MR. C. E. EDWARDS at Appleby-Frodingham.

MR. ASHLEY S. WARD, who has been Chairman of Thos. W. Ward, Ltd., since 1941 has resigned from that office. The Company will not wholly be deprived of his specialised knowledge and experience as he remains a Director of the Company and will act in an advisory capacity. In recognition of Mr. Ashley Ward's many years of splendid service the Company have appointed him President as from January 4th, 1950.
MR. GEORGE WOOD has been appointed Chairman and MR. FRANK R. STAGG Deputy Chairman; MR. H. W. SECKER, O.B.E. and MR. J. WALTON become Joint Managing Directors; and MR. ARNOLD CARR, hitherto in charge of London area operations, is to transfer his activities to Sheffield for other important work. MR. F. W. ROBINSON, a Local Director; has been appointed to the full board of the company and will take charge of London area interests vice Mr. Arnold Carr.

MR. E. K. HUGHES, Commercial Manager and a Special Director of the Whitehead Iron and Steel Co., Ltd., has succeeded the late Mr. Wm. Brown in London. MR. T. G. ROLGER, of the Consett Iron Co., Ltd., has been appointed Commercial Manager. The Company's Birmingham office will be in charge of MR. R. MILLS, who will have MR. D. R. GREENHOUGH as assistant.

New Year Honours List

IN the New Year Honours List, published as a supplement to the *London Gazette*, a number of scientists, industrialists and technicians were honoured, amongst them the following:—

K.B.E.—F. BRUNDRETT, Esq., Chief of the Royal Naval Scientific Service.

C.B.—S. S. HALL, Esq., Director-General of Technical Development (Air), Ministry of Supply.

C.B.E.—J. R. BEARD, Esq., Senior Partner, Merz & McLellan; J. McF. McNEILL, Esq., Managing Director, John Brown & Co., Ltd., Glasgow; J. MORRISON, Esq., Director and Shipyard Manager, Harland & Wolff, Ltd., Belfast; F. M. OWNER, Esq., Chief Engineer, Engine Division, Bristol Aeroplane Co., Ltd.; H. RUSSELL, Esq., lately Director of Aluminium House Production, Ministry of Supply; K. T. SPENCER, Esq., Deputy Director, Aircraft Research and Development (Technical), Ministry of Supply; PROFESSOR A. M. TYNDALL, Chairman, National Physical Laboratory Executive Committee.

O.B.E.—R. M. CLARKSON, Esq., Assistant Chief Engineer (Aircraft) and Chief of Aerodynamics Department, De Havilland Aircraft Co., Ltd.; E. ELLIS, Esq., Joint Manager, Repairs Department, J. I. Thorneycroft & Co., Ltd., Southampton; G. B. S. ERRINGTON, Esq., Chief Test Pilot, Airspeed, Ltd., Christchurch; T. GOLDING, Esq., Manager, Electrical Department, W. H. Allen, Sons & Co., Ltd., Bedford; E. GRIFFITHS, Esq., Senior Principal Scientific Officer, Department of Scientific and Industrial Research; J. G. PEARCE, Esq., Director, British Cast Iron Research Association; J. R. PEARSON, Esq., Director and Factory Manager, Vauxhall Motors, Ltd., Luton; J. E. RICHARDSON, Esq., Engineering Manager, Vickers-Armstrong, Ltd., Barrow-in-Furness; F. F. J. B. ROBERTS, Esq., Director, Basic Slag, Ltd.

M.B.E.—G. H. ALDRED, Esq., Chief Engineer, British Ropes, Ltd., Doncaster; A. S. BROWN, Esq., Manager, Electric Department, Clarke Chapman & Co., Ltd., Gateshead; L. C. CLARKSON, Esq., Works Director, Raleigh Industries, Ltd., Nottingham; A. HASELDEN, Esq., General Works Manager, Ford Motor Co., Ltd., Dagenham; A. J. HEWITT, Esq., Superintendent of Gas Turbine Aero Engine Development, Armstrong-Siddeley Motors, Ltd., Coventry; E. N. HILEY, Esq., General Secretary, National Brass Foundry Association; D. KING, Esq., Chief Armament Designer, De Havilland Aircraft Co., Ltd.; K. B. LING, Esq., Superintendent, Standard Telephone and Cables, Ltd., Treforest; W. G. MASTERS, Esq., Senior Technical Officer, Royal Mint; N. H. SCARTH, Esq., Manager, Engine Department, Yarrow & Co., Ltd., Glasgow; H. C. STEVENS, Esq., Heavy Forge Manager, William Jessop & Sons, Ltd., Sheffield; J. B. STEVENSON, Esq., Works Director, E.M.I. Factories, Ltd., Hayes, Middlesex; J. TAYLOR, Esq., Dockmaster, Cammell Laird & Co., Ltd., Birkenhead; G. W. WARREN, Esq., Physicist, Research Laboratories, General Electric Co., Ltd.; C. J. WHITTALL, Esq., in charge of Admiralty Division, Elliott Bros. (London), Ltd.; T. H. WILLIAMS, Esq., Mill Manager and Chief Roll Designer, Cargo Fleet Iron Co., Ltd., Middlesbrough; H. W. WILSON, Esq., Admiralty Liaison Engineer, General Electric Co., Ltd., Birmingham.

Reviews of Current Literature

ATLAS OF ISOTHERMAL TRANSFORMATION DIAGRAMS

Special Report No. 40 by Thermal Treatment Sub-Committee of the British Iron and Steel Research Association, published by the Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1; price 15s. to members of the Institute and 25s. to non-members.

CONTROL over the results of heat treatment of steel has been extended in recent years by the study of "S-curves" or isothermal transformation diagrams. These curves show what happens to the structure of a particular steel when it is held at a certain temperature after rapid cooling from above the critical temperature, which is usually in the range of 700°–800° C. They show the varying effects on the structure of holding or "soaking" at various temperatures for different periods of time. Below a temperature known as the MS point the martensitic transformation is instantaneous, although the degree of transformation can be controlled by suitable choice of quenching temperature. All transformation temperatures vary according to the composition of the steel.

For example, if a 3% nickel steel (En 21) is quickly cooled from above 700° C. to about 400° C. and held at that temperature, transformation of the austenite begins after about 15 seconds, and the amount transformed is respectively 10%, 50%, 90%, and 100% after about 30, 50, 100, and 400 seconds. But if the soaking temperature is 350° C. transformation begins after 35 seconds, and the corresponding periods for 10, 50, 90 and 100% are about 75, 150, 300 and 900 seconds. The MS point for this steel is 310° C.

For this particular steel, numbers of detailed observations have been made at 13 temperatures, ranging between 350° C. and 700° C. The S-curves plotted from these observations correspond to the five stages of transformation mentioned above. They show the time of "soaking" required at each temperature on the curves for each stage to be reached.

The data has been collected by the Thermal Treatment Sub-Committee of the British Iron and Steel Research Association and represent the results of collaborative work by a number of industrial laboratories and provides the first authoritative published collection of S-curves for the main types of wrought steel in current use in this country. The Atlas contains S-curves for 24 En steels of the British Standards Institution's En series detailed in B.S. 970: En 12–19, 21, 23–26, 28, 30, 31, 40, 42, 44, 45, 47, 56, 100 and 110. Each diagram is accompanied by a data sheet which records the compositions of the steel, its previous treatment, the hardness of the transformation products obtained at various temperatures, photomicrographs and general comments which include reference to the hardenability of the steel.

The text of the report outlines the principles underlying the construction of isothermal transformation diagrams and the experimental procedure used for the determination of the curves, and discusses the results and their application.

It is pointed out, however, the main purpose of isothermal transformation diagrams is to provide a basis for appreciating the behaviour of steels when quenched in sections which do not harden completely, rather than to give data for the design of heat treatment schedules. They are, in fact, often regarded as offering no more than a general indication of the transformation behaviour of a given steel, but the special care taken in this investigation to check the representative nature of the steels and the confirmation of the curves by independent laboratories justifies their more confident use.

The report is bound in stiff covers and the diagrams are printed on good quality paper suitable for pencilling in additional curves. Curves traced on transparent isothermal transformation test charts may be conveniently superimposed on the printed diagrams. Suitable charts have been designed by the Thermal Treatment Sub-Committee and are available from Pawson and Brailsford, Ltd., Mulberry Street, Sheffield.

METAL RECTIFIERS

By H. K. Henisch, 1949, pp. 155. One of the monographs on the Physics and Chemistry of Materials; general editors: B. Chalmers, Willis Jackson, H. Fröhlich, N. F. Moth. Published by Geoffrey Cumberlege, Oxford, at the Clarendon Press; price 15s.

THIS book fulfils a need which has existed since Mott and Schottky developed their theories of rectification at the boundary between a metal and a semi-conductor. It places side by side theory and experiment, and will be most useful to persons engaging on rectifier research and development. No attempt is made to present extensive data on the operation and application of rectifiers, since these are available in manufacturers' leaflets and in the technical press.

The book falls roughly into two parts. The first consists of a description of the methods of manufacture of the three main plate rectifiers—selenium, copper oxide and sulphide rectifiers—followed by a more detailed chapter on their electrical characteristics. The germanium catswhisker rectifier is also mentioned, but not the silicon one. The electrical characteristics described include current-voltage curves, rectifier self-capacity, creep and ageing effects. A short and useful chapter on measuring techniques follows. The second half of the book is devoted to an exposition of present-day theories of rectifier action. Since these theories require a knowledge of semi-conductors in general a brief outline is given of the various types of impurity centre and of such phenomena as conduction by electrons and positive holes, intrinsic and impurity conduction. All this is fitted into the band theory of solids, but it was perhaps unnecessary to dwell at any length on the now well-known picture of energy bands, since the serious student will no doubt consult the standard textbooks on the subject.

The development of the theories of rectification, which apply to the various types of barrier, is carried out in a clear and concise manner and is followed by a brief comparison with experimental results. This comparison strikes one as being rather short in view of the large mass of experimental data available. Although Dr. Henisch quite rightly states that the theories only apply to special laboratory-made rectifiers, he does not, for example, point out that even after fitting the curves at very small potentials, Schottky's current-voltage characteristics only agree with the experimental ones up to a few tenths of a volt.

A short chapter on rectifier operation deals with rectifier efficiency and rectifier breakdown, this being defined as the permanent destruction of the rectifier and

consequently ascribed to thermal effects. This may be slightly misleading, since by breakdown is often meant the rise in current which occurs as a certain blocking voltage is exceeded, and this phenomenon is certainly not due to heating effects but causes them.

The book ends with some notes on the directions which future developments will probably take, and a very extensive bibliography is appended, consisting of about 500 references which range over the whole field of semi-conductors. This is a most useful feature, since no review article containing such a bibliography has been written of late.

K. W. P.

MANUALE DI FONDERIA D'ALLUMINIO. TEXTBOOK OF ALUMINIUM CASTINGS

By Carlo Panseri. Milan: Ulrico Hoepli Editore, 1949; paper covers, 2,500 lire, cloth bound, 3,300 lire. 8vo, 476 pp., with 130 figs., 194 tables, 3 appendices (in Italian).

THE author of this work, Prof. Carlo Panseri, is Director of the Light Metal Research Institute, Montecatini-Sava Foundation, Milan—Via Della Posta 8–10. He is well known for his work in this field, and is author of a companion volume on Aluminium and its Alloys. Italy may well take her place among the leading nations in aluminium technology and research, not only for the fundamental work undertaken at this Institute, but also on account of the fact that the Montecatini concern have, though with a relatively late start, specialised in this field.

The book deals rather fully both with aluminium and its alloys, of which a list of 884 is given in the first appendix. The title is indeed hardly adequate, for much more than casting of aluminium is covered. Some interesting statistics are given in Chapter 1 on aluminium production and consumption, with particular reference to Italy's share. In 1947 total world output is given as 1,100,000 tons, of which 23% was European, as compared with the peak year (1943) when it was 1,935,000 tons, with a European percentage of 27%. Although Chapter 2 is headed "General characteristics of pure and commercial aluminium," the author is careful to emphasise that aluminium as a chemical element, perfectly pure, is at present impossible to achieve—with all our refined modern resources.

The next three chapters (3–5) treat respectively of the theory of light alloys, the solidification of aluminium and its alloys (crystallography) and contraction (segregation) and gaseous solubility. Then follow the important sections on heat treatment of light alloys, with the various types of annealing, ageing, etc.; alloy properties; and, of special interest, physico-mechanical tests of castings. These latter, relating to hardness and surface conditions generally, resistance to fatigue, tensile strength and other qualities, are to-day of the utmost importance. The author appears to be well up to date here; and there is little doubt that the Italians are abreast of modern research. It is a little strange or unusual, however, that in this fairly exhaustive treatise there is no bibliography, and little or no reference to the work of others; though there is a general acknowledgment at the beginning.

The next Chapter (9) on methods of metallographic and analytical control is also of particular interest. It includes micrographic examination, but not very adequately; and a serious omission would seem to be that little or no reference is made to the value and significance of electrolytic polishing in connection with micrographic research, as developed, for example, by Jacquet in the first volume of his work on "*Le Polissage Electrolytique*" which is wholly devoted to aluminium, magnesium and light alloys. It is, of course, difficult to include a full account of everything in a vast and ever widening field. Possibly it is here that a judicious selection of literature references might prove helpful; especially if just a little more than the title could be included in those references.

The author now arrives, in Chapter 10, at the real essence of his work, and describes in detail, with the help of numerous excellent diagrams, the various types of furnace, heating, temperature measurement and control; and in Chapter 11, the actual melting, de-oxidising and de-gassing and alloying processes. Moulding and and related operations, in sand, etc., including frame casting and under pressure, finishing or fettling, and so on, are in Chapters 12–15.

The remainder of the book, Chapters 16–20 is mainly occupied with choice of materials for alloy casting, characteristics of binary alloys (Al-Cu, Al-Si, Al-Mg and Al-Zn), and the most suitable alloys for castings for (*a*) general use; (*b*) corrosion-resistant; and (*c*) special purpose castings. The corrosion-resistant type are of course of great industrial and also architectural importance, as well as in naval and general shipping construction. Special uses include the heat-resistant, anti-friction and electrical types. As already mentioned, the first appendix contains no less than 884 alloys, with trade names and compositions, and shows with what rapidity this department of light metal technology is advancing. There is no index, but a fairly detailed contents table.

W.G.C.

THE NON-FERROUS METAL INDUSTRY IN GERMANY—1939–1945.

Compiled by Leslie Aitchison, M.Sc., D.Met., F.R.I.C., F.R.Ae.S., M.I.Mech.E. and Voya Kondic, B.Sc., Ph.D., and published for the Ministry of Supply and the Technical Information and Documents Unit of the Board of Trade by H.M. Stationery Office, price 3s. 6d., at any of the Stationery Office branches, or 3s. 9d. by post.

BETWEEN November, 1944 and May, 1947, non-ferrous metal plants and research centres in Germany were visited by about 150 teams of British and American experts, who issued numerous reports on their investigations. In addition, a large number of documents dealing with production figures, research activities, manufacturing processes, plant drawings, etc., were brought back to this country. Considerable information has accumulated and is available as a result, but reference to any particular subject had been a rather lengthy process, since the information obtained had not been correlated. This position has now been rectified by the publication of this B.I.O.S. Survey No. 23, which gives a general review of the German non-ferrous metal industry during the period noted and greatly simplifies the investigation of particular sections of the industry.

The Survey is divided into four main parts, dealing with light metals, base metals, rare metals and precious metals. The first three parts are sub-divided into sections on smelting and refining, casting, wrought fabrication and finishing, alloys and applications. Each part carries a list of references to the reports surveyed, and a general classified bibliography of relevant reports and documents available in the United Kingdom. Also included is a full index and cross-reference system.

METALLURGICAL DIGEST

Investigations into Corrosion-Resisting Cast Chromium-Nickel Steel with Special Reference to the Sigma Phase

THE influence of heat treatment on the formation of σ-phase in a corrosion-resisting chromium-nickel-molybdenum cast steel of type 20-14-4 (A.I.S.I. 317) was investigated in three series of tests in the Sulzer metallurgical laboratories as part of research work in progress on corrosion-resisting cast alloy steels. The material tested, which was melted in basic electric furnaces in the normal course of foundry operation, had the following chemical composition :—

C	Si	Mn	P	S	Cr	Ni	Mo
0·12	0·21	0·56	0·03	0·01	20·67	11·54	4·11

The testing methods used to investigate the influence of heat treatment on σ-formation were the determination of notch toughness and Brinell hardness ; examination of the microstructure ; and measurement of the magnetic permeability. The first series of tests was conducted on specimens of the following types and treatments :—

Cast and forged, without heat treatment ; annealed at 1,050° C. for 4 hours, cooled in oil, air and furnace ; and annealed at 1,150° C. for 4 hours, cooled in oil, air and furnace. Mechanical and magnetic tests are summarised in Tables I and II.

It is clear from Table I that this material has a high σ-phase content in the cast state and is consequently brittle. Annealing at 1,050° C. suffices to improve the toughness only slightly. When the annealing temperature is raised to 1,150° C., however, the σ-phase disappears completely after quenching in oil, and the toughness of the material is therefore high. On the other hand delayed cooling in the furnace is sufficient to make the steel completely brittle again as a result of σ-phase formation, showing that the separation of the σ-phase must take place very rapidly in certain temperature ranges. Similar processes take place in the forged metal, although the absolute values of the notch toughness are higher than with the cast material.

The second series of tests was concerned with the influence of tempering temperature on the formation of σ-phase. The investigations covered cast and forged specimens which were quenched in oil at 1,150° C. after annealing for 4 hours, were then tempered for a period of 4 hours between 500° and 1,050° C. and subsequently cooled. The results of mechanised and magnetic tests carried out are given in Tables III and IV for the specimens cooled in air after tempering.

With a tempering treatment lasting 4 hours, the separation of the σ-phase is noticeable from 550° C. onwards. At 850° C. the ferrite has transformed almost completely to σ-phase. In this condition the steel is practically non-magnetic. With a further rise in the tempering temperature beyond about 950° C. the σ-component is reduced again as the result of the formation of new ferrite. In the temperature range of this new ferrite formation the influence of the cooling rate mentioned above makes itself clearly manifest, as with delayed cooling in air, and even

TABLE I.—NOTCH TOUGHNESS, BRINELL HARDNESS AND QUANTITY OF σ-PHASE IN CAST SPECIMENS OF CHROMIUM-NICKEL-MOLYBDENUM STEEL (20-14-4), AFTER VARIOUS HEAT TREATMENTS

Condition and heat treatment	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15	σ-phase % *
As cast	0·7– 0·8	197–207	94·5
1,050° C./4 hrs./oil-cooled	3·7– 4·4	184–187	Not determined
1,050° C./4 hrs./air-cooled	0·8– 1·1	184–193	" "
1,050° C./4 hrs./furnace-cooled	0·3– 0·4	184–200	" "
1,150° C./4 hrs./oil-cooled	23·0–28·0	170–172	0 "
1,150° C./4 hrs./air-cooled	18·4–19·2	160–164	15·5
1,150° C./4 hrs./furnace-cooled	1·3– 1·5	170–179	92·5

* Expressed as a percentage of the ferrite quantity in the specimen quenched in oil at 1,150° C.

TABLE II.—NOTCH TOUGHNESS AND BRINELL HARDNESS OF FORGED SPECIMENS OF CAST CHROMIUM-NICKEL-MOLYBDENUM STEEL (20-14-4), AFTER VARIOUS HEAT TREATMENTS

Condition and heat treatment	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15
Forged—13 mm.	14·3–15·3	186–189
1,150° C./4 hrs./oil-cooled	39·5–39·6	165–168
1,150° C./4 hrs./air-cooled	20·9–21·3	170–180
1,150° C./4 hrs./furnace-cooled	9·3–11·1	187–197

TABLE III.—NOTCH TOUGHNESS, BRINELL HARDNESS AND QUANTITY OF σ-PHASE IN CAST SPECIMENS OF CHROMIUM-NICKEL-MOLYBDENUM STEEL, AFTER VARIOUS HEAT TREATMENTS

Treatment—1,150° C./4 hrs./oil and tempered at :	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15	σ-phase % *
500° C./4 hrs./air	26·9–32·1	167–181	4·0
550° C./4 hrs./air	21·5–21·7	176–186	18·0
600° C./4 hrs./air	13·4–14·4	174–189	26·5
650° C./4 hrs./air	7·2–10·0	174–177	58·5
700° C./4 hrs./air	2·1– 4·8	190–193	86·5
750° C./4 hrs./air	2·0– 3·7	172–191	91·5–93·0
850° C./4 hrs./air	1·7– 2·0	179–180	97·0
1,050° C./4 hrs./air	1·8– 2·7	182–187	81·0

* Expressed as a percentage of the ferrite quantity in the specimen quenched in oil at 1,150° C.

TABLE IV.—NOTCH TOUGHNESS AND BRINELL HARDNESS OF FORGED SPECIMENS OF CAST CHROMIUM-NICKEL-MOLYBDENUM STEEL AFTER VARIOUS HEAT TREATMENTS

Treatment—1,150° C./4 hrs./oil and tempered at :	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15
500° C./4 hrs./air	32·2–35·1	169–172
550° C./4 hrs./air	27·6–29·0	176–182
600° C./4 hrs./air	17·1–17·6	185–187
650° C./4 hrs./air	14·0–14·9	175–179
750° C./4 hrs./air	10·8–11·2	170–179
850° C./4 hrs./air	11·1–12·0	182–185
1,050° C./4 hrs./air	8·3– 9·1	161–170

From *Sulzer Technical Rev.*, 1949, No. 2, pp. 1–8.

TABLE V.—NOTCH TOUGHNESS, BRINELL HARDNESS AND σ-FORMATION IN CAST SPECIMENS OF CHROMIUM-NICKEL-MOLYBDENUM STEEL (20-14-4) AFTER VARIOUS HEAT TREATMENTS

Treatment—1,150° C./4 hrs./oil and tempered at :	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15	σ-phase % *
550° C./4 hrs./air	21·5–21·7	176–186	18
550° C./24 hrs./air	13·3–13·5	175–177	19·5
550° C./48 hrs./air	12·0–12·5	177–181	32·5
550° C./100 hrs./air	9·3–10·4	177–177	49·5
750° C./30 mins./air	4·3–10·0	171–174	66·5–67·0
750° C./2 hrs./air	4·5– 6·1	174–183	86·0–88·5
750° C./4 hrs./air	2·0– 3·7	172–191	91·5–93·0

* Expressed as a percentage of the ferrite quantity in the specimen quenched in oil at 1,150° C.

TABLE VI.—NOTCH TOUGHNESS AND BRINELL HARDNESS OF FORGED SPECIMENS OF CAST CHROMIUM-NICKEL-MOLYBDENUM STEEL (20-14-4) AFTER VARIOUS HEAT TREATMENTS

Treatment—1,150° C./4 hrs./oil and tempered at :	Notch toughness V.S.M. 10 mkg./cm.²	Brinell hardness H 5/750/15
550° C./4 hrs./air	27·6–29·0	176–182
550° C./24 hrs./air	15·7–17·9	178–180
550° C./48 hrs./air	14·3–18·0	178–186
550° C./100 hrs./air	13·3–14·1	174–178

more so in the furnace, the σ-phase is again produced from the ferrite.

When the σ-phase is formed, the steel suffers a considerable loss in toughness, so that marked embrittlement results, particularly in the critical temperature range between about 700° and 1,050° C. The correlation is such that the notch toughness can be regarded, at least from a quantitative viewpoint, as a means of assessing the extent of σ-formation.

The third series of tests was intended to establish the course of the σ-formation in ferrite in time at tempering temperatures of 550° and 750° C. All the test pieces were quenched in oil at 1,150° C. before tempering. The results of the mechanical and magnetic tests are given in Tables V and VI.

At 550° C., it will be noted, the σ-formation is still relatively slow, so that it is only after about 100 hours that 50% of the original ferrite has been transformed to σ-phase. At 750° C. separation takes place more rapidly. After a tempering period of 20 minutes, more than 60% of the ferrite has been changed, and after 4 hours more than 90%. This dependence on time and temperature makes it appear that the separation of σ-phase from the ferrite is a diffusion process.

These test results reveal that steel of 20-14-4 composition is not, in general, suitable for use in castings, or, at any rate, not in cases where it cannot be submitted to suitable heat treatment.

Dephosphorisation of Iron Ore

By C. G. Granström

A METHOD developed for dephosphorising iron ore was patented as long ago as 1870, but the primary object of the development was the removal of phosphates from the ore with a view to using the phosphates. As a means of improving the iron ore a method of dephosphorising, by leaching with acids, was suggested by G. A. Granström in a patent granted in 1897. The possibilities of this method, however, were not investigated until 1937–40. Laboratory experiments and tests were then made in a small experimental plant, and, among other results it was observed that phosphorus was precipitated in the product if hydrochloric acid was used as a solution agent in the extraction.

The problem was taken up on a bigger scale, in 1941, at the Grängesberg export ore field, and favourable results were obtained with nitric acid. An experimental plant was built, in which highly concentrated iron ore slurry containing about 0·06% phosphorus was treated with nitric acid and a desulphurised slurry is produced, containing more than 71% iron and below 0·01% phosphorus. The consumption of acid, however, was rather high.

Since 1945 investigations have also been carried out by a research committee of Jernkontoret in close contact with the work at Grängesberg and interesting results have been obtained concerning the influence of the size and character of the ore grains. Based on the experience gained a new full-size plant has been built at Grängesberg for an annual production of 30,000 tons of dephosphorised iron ore concentrates. The plant was just recently started and the results so far seem very promising.

Features of the Grängesberg dephosphorisation method are the use of nitric acid as a solution agent and the limited percentage of water used in the extraction slurry. It is believed that this method will ensure the supply of pure iron ore for the production of sponge iron in Sweden, although more experience must be gained with regard to the treatment of ores other than those from the Grängesberg mines.

From *Jernk. Annal.*, 1949, **133**, No. 9, pp. 323–367.

On the Stress Corrosion Resistance of Aluminium Alloys

By G. SCHIKARR and G. WASSERMANN

SOME results are given of a co-operative research commenced in 1943 on the stress corrosion resistance of aluminium alloys exposed to natural weathering in different localities (marine, industrial, urban, rural) in Germany. The results are of a preliminary nature as the work was interrupted by the war and some of the material and records have been lost.

Specimens cut from sheets were bent and the ends riveted together to form the well-known loop test pieces. The alloys tested included Al-Cu-Mg clad and unclad, Al-9% Mg (half-hard), Al-Zn-Mg (quenched in water), Al-Zn-Mg (specially heat treated to reduce sensitivity to stress corrosion) and Al-Zn-Mg alloys with addition of vanadium and chromium. The results of the natural exposure tests were supplemented and compared with results of laboratory tests in which similar loop specimens were partly immersed in 3% sodium chloride solution.

In view of the frequently non-uniform distribution of the times taken by the specimens in any one set to fracture, it was found preferable to express the results of the tests by the time taken for half the specimens of a particular set to fracture than by the mean life of the specimens in a set.

Comparing the results of natural exposure tests with those of the laboratory tests, a qualitative agreement was found to exist, but this did not allow any quantitative deductions to be made from the laboratory tests as to the weathering resistance of the stressed aluminium alloys.

From *Zeits f. Metallk.*, June, 1949, 203–205. Reproduced from *Light Metals Review*, 1025.

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS

JANUARY, 1950 Vol. XLI No. 243

The Application of Electrical Strain Gauges and Brittle Lacquers to Engineering Design

By J. S. Caswell, M.Sc.
(*Engineering Department, University College, Swansea*)

An important advance in the methods of engineering design is marked by the rapid developments which have taken place, during the last few years, in the use of electrical resistance strain gauges and brittle lacquers for the determination of stresses in engineering structures and components. A general account of the two methods is given and attention is drawn to their complementary nature.

ENGINEERING design may be defined as a process of discovering and providing information for the building of machines and structures which are correct in form and detail, and properly suited for their purpose. Correctness of form and detail is not easily obtained because the knowledge and experience required is generally incomplete. Mechanical and structural design is founded mainly on the following :—

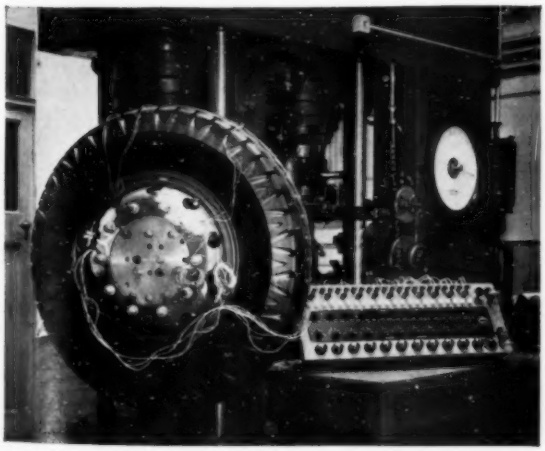
(*a*) Knowledge of the forces to which a machine or structure may be subjected.

(*b*) Knowledge of the properties of engineering materials and the theory of elasticity.

(*c*) Skilful guesswork which is based on practical experience.

Over the past fifty years much progress has been made in (*a*) and (*b*) and, as a result of increased experience, the quality of the guesswork has been correspondingly improved. Consequently, the approach to correct design has been steadily improving and, over a wide range of engineering work, a very close approach to ideal design has become possible. There is, however, a wider range of design in which a high degree of accuracy cannot be obtained, because the magnitude and the nature of the loading, and also the complex variation of internal stresses are not adequately known. For many of these difficult problems of design, where the loading is steady and there is wide latitude in the amount of material that may be used, the risk of failure can be largely eliminated by using high factors of safety. Moreover, the ductile qualities of some materials ensure that high local stress can be relieved by local yielding, and the general stress conditions are thereby improved. This procedure involves increased weight and cost of material, both undesirable features for many aspects of design.

When the loads are dynamic in character, uncertainty regarding the internal stress values may be fraught with heavy risk of failure of a machine or structural part. A local, isolated high value of stress, operating on a minute area of cross-section in a cyclic manner, may be sufficient to initiate a fatigue crack and propagate spreading of the crack until complete failure results. It is the unknown high values of local stress, under dynamic loading, that sometimes defeat the best efforts of designers. The complexity of form of machine or structural parts adds further difficulty, by rendering mathematical methods of stress analysis inadequate or unreliable. In these cases, approach to correct design is made by using a series of approximations for which strength equations can be evolved, and these equations may be modified by the use of constants found by empirical methods. This form of approach is very common, and it governs a great deal of design in the construction of ship machinery and other plant covered by commercial and industrial insurance, but a good designer, while guided by this form of approach, is fully aware of its serious limitations.



Courtesy of the Motor Industry Research Association

Fig. 1.—Set-up for the measurement of strain in road wheels under static loading.

Courtesy of the Motor Industry Research Association

Fig. 2.—Measuring the local static bending stresses in a crankshaft using wire resistance gauges of ¼ in. gauge length.

The Investigation of Internal Stress

This uncertain quality of the conventional form of design method is the chief underlying cause of a very large number of service failures, and, unfortunately, these are sometimes accompanied by serious losses of life and money. The increasing tempo of engineering development, and the paramount need for safe and uninterrupted service, have created an urgent demand for more reliable methods of design, the urgency being centred on the need for accurate knowledge of the magnitude and precise location of dangerous internal stresses. Notwithstanding the excellence of the theoretical methods of approach, it is the methods of experimental stress analysis which are pre-dominant in meeting this need, and they have brought about a completely new approach to complex engineering design. For many years the need was, and is still being, very effectively met by the experimental methods of photo-elasticity which were developed in a classical manner by the late Professor E. G. Coker, F.R.S. and the late Professor L. N. G. Filon, F.R.S. Comprehensive testing of full-scale prototypes has also played an important part in the investigation of internal stress under service conditions.

All the early work in photo-elasticity was confined, as much of the present work is, to the analysis of two-dimensional stress systems. The models used vary in form in two dimensions, but they have a uniform third dimension, which is equal to the thickness of the sheet of transparent photo-elastic material used in making the model. In some cases, two or more thicknesses of sheet may be involved. Consequently the range of application of this two-dimensional method must be carefully limited to those cases which are reasonably representative of the critical system of stress in the three dimensional machine or structural part. In recent years the photo-elastic method has been extended to the analysis of three dimensional stress systems. Scale models of complex engineering parts in optically sensitive plastics can be made, and the applied loads produce stress conditions which more closely represent the stress conditions in the real machine part. The limitations of this method lie in the difficulty of producing satisfactory models, but the method has greatly increased the value of the photo-elastic method of approach for obtaining correct design.

In view of the difficulties of simulating real conditions in the photo-elastic method, it is easy to understand the preference for testing full size prototypes of machine and structural parts. Tests of these parts usually proceed to destruction or to a stage at which failure at some vital point becomes evident. The information gained is largely confined to the point of failure, and the part tested is usually unsuited for further use.

During the war years, there was accelerated development of two new and important methods of approach to the solution of design problems—viz.: (*a*) the use of electrical resistance strain gauges, and (*b*) the use of brittle lacquers. These methods provide powerful aid for the designer because they have made it possible for him to do what was hitherto impossible—viz.: to measure stresses produced by dynamic forces in high-speed machinery and vibrating structures. Each method has its special field of application, and both methods have important complementary applications which make stress investigation more precise and rapid. These methods are discussed in general outline below. Their application for design purposes expanded rapidly under the impetus of war requirements, and the unique advantages of the methods have made them an indispensable part of drawing office procedure in many vital aspects of high quality engineering design.

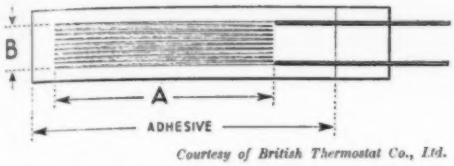
Method I—The Electrical Resistance Strain Gauge

This method is based on the fact, first noted by Lord Kelvin, that when a wire is elongated, its electrical resistance is changed. If the elongation occurs within the elastic range of the material, there is a linear relation between the elastic strain and the change of resistance. Consequently, when a suitably insulated wire is rigidly attached to the surface of a machine or structural part, the change of strain accompanying the change of stress, produces a change in the electrical resistance of the wire; the change of resistance can be accurately measured and expressed in terms of the change of stress. In order to obtain adequate change of resistance without extensive longitudinal bonding of the wire, and also for the purpose of confining the strain changes to a small area, a length of Ni-Cr or Cu-Ni wire about 5 in. long and about 0·001 in. diameter is arranged in a series of loops as shown in Fig. 3.

A small amount of transverse strain is taken up at the loop ends, and a suitable correction can be made for this when the gauge is calibrated. The looped wire, already under slight strain, is bonded between thin strips of paper, and the ends of the wire are spot-welded to fairly stout tinned metal strips to which connecting leads can be soldered. The gauges have assigned resistance values ranging from 100–2,000 ohms, and the accurate value of the resistance is marked on each gauge. The length over the loops is the gauge length, and it has the meaning commonly associated with mechanical testing; the length ranges from about 1½–½ in. for general purposes, but may be as small as $\frac{1}{16}$ in. for gauges used in confined spaces or at points of intense stress concentration.

The Use of the Gauge and the Calculation of the Stress

The gauges can be attached to machine or structural members by the simple process of sticking them on with the aid of a good adhesive. Alternatively, a self-adhesive type may be used, requiring only a dip in



Courtesy of British Thermostat Co., Ltd.

Fig. 3.—Strain gauge. (A—gauge length, B—gauge width).

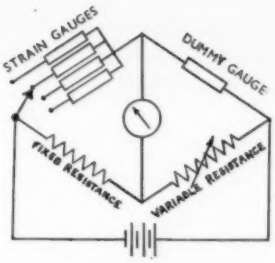
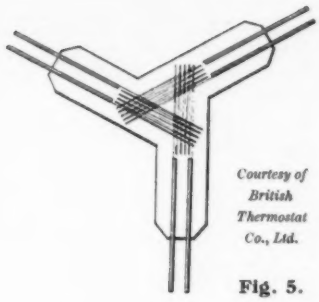


Fig. 4.—Wheatstone bridge set-up.

Fig. 4.



Courtesy of British Thermostat Co., Ltd.

Fig. 5.—Delta strain gauge for the analysis of bi-axial stress systems (60° included angles).

Fig. 5.

acetone and quick attachment to the surface. The surface should be smooth and thoroughly clean, and care should be taken to ensure good bonding with the surface over the whole under-surface of the gauge. The gauges should be protected from damp and disturbance while setting, and afterwards covered with a damp-proof material—e.g , vaseline, varnish, rubber solution, wax, or one of the specially prepared proprietary insulators. Binding with insulating tape is a further precaution required in some cases. The gauge is now in a condition to respond to change of stress, and the linear relation between the unit change of resistance and the unit strain can be simply expressed by a value termed the gauge factor, as shown below. (The method of calculating the stress change accompanying a measured change of resistance, is also shown).

$$\frac{\delta R}{R} \Big/ \frac{\delta l}{l} = \phi$$

where: δR = change of resistance, in ohms.
R = resistance of the strain gauge, in ohms.
δl = length change over the gauge length l, in inches.
l = gauge length, in inches.
ϕ = gauge factor (a value which is supplied by the manufacturers of the gauge).

and since the stress associated with the strain is:—

$$\sigma = \frac{\delta l}{l} E$$

where: σ = the change of stress, in tons/sq. in., and
E = the modulus of elasticity, in tons/sq. in.

therefore,

$$\frac{\delta l}{l} = \frac{\sigma}{E}$$

and

$$\phi = \frac{\delta R}{R} \Big/ \frac{\sigma}{E}$$

thus giving the change of stress

$$\sigma = \frac{E \ \delta R}{R \ \phi} = \text{Constant} \times \delta R$$

Observe that the constant is determined from the initially known values of E, R and ϕ, and consequently, when the value of the change of resistance δR has been measured, the change of stress may be calculated. Autographic recording apparatus has been produced for showing directly the value of the change of stress, and it is worth noting that it is only in so far as electronic engineers are able to produce reliable and easily handled instruments, that the practising civil or mechanical engineer will place confidence in this method of stress measurement.

For many purposes where static or almost static stresses have to be measured, the simple form of Wheatstone bridge set-up may be used, with the working gauge forming one arm of the bridge (Fig. 4). Balance conditions before and after the application of stress are obtained, and the change of resistance in the working gauge is found from the final bridge-arm ratios. By the use of a suitable switching arrangement, a number of separate gauges located at different parts of a structure may be coupled to the same bridge or to separate bridges, incorporated in a switching unit. Since temperature changes produce slight expansion or contraction of a structural part and the gauge attached to it, it is necessary to compensate for the effects of this change; and this may be done by attaching another gauge to a loose unstressed bar of structural material similar to that under test. This gauge may then be used to obtain zero balance of the Wheatstone bridge before the active gauge having the same resistance, is used.

The customary laboratory method of setting up a Wheatstone bridge circuit involves the use of numerous pieces of apparatus, and one of these is a highly sensitive and delicate galvanometer. This apparatus and the method of using it are quite unsuited for engineering field work, but manufacturers, by making use of electronic amplifying circuits, are producing robust portable instruments suitable for field work. The electrical connections in the field are usually confined to simple couplings to the strain gauges and to a simple power source.

Field instruments with circular or roller charts for permanent ink records are obtainable for recording non-cyclic stresses, and simple photographic apparatus can be used for recording cyclic stresses. Observations of stress change can be made rapidly—e.g., 144 strain gauges attached to different parts of an 80-ft. span all-welded roof truss, under test loads, could be scanned within a period of two minutes, but a special recorder, made by Messrs. Savage & Parsons, Ltd. for stress investigation on large aircraft, scans 600 gauges in about eight seconds.

Strain and Stress

In a simple uniform stress system—e.g., as in the mid-length of a tie bar, a single strain gauge suffices for the measurement of stress, but in complex stress systems, a single gauge registers only the net effect of the surface strains resolved in the direction of the gauge. Consequently, the directions of the principal strains and stresses cannot be found without the measurement of strains by gauges set in two other directions. Simple gauges may be used, but this set-up usually occupies too large an area, and it is more convenient to use the rosette type of gauge; this being merely an arrangement of three independent looped resistance wires, as used for gauges, in the form of a triangle with convenient angles. Such a gauge is mounted in the manner described earlier

and, on measuring the strains produced in the separate elements of the gauge, three simultaneous equations can be written down and solved to determine the values of the principal strains and stresses. This type of gauge is shown in Fig. 5.

The preceding notes give a general outline of the electrical strain gauge method for use in its simplest form. With the aid of a portable strain indicator, a switching unit for bringing different gauges into the bridge circuit, stress investigations over a wide range of practice can be carried out with ease and accuracy. It should be observed, however, that this simple form of stress measurement can only be applied to those cases where the stress values are steady or change very slowly.

The Measurement of Rapidly Changing Stress

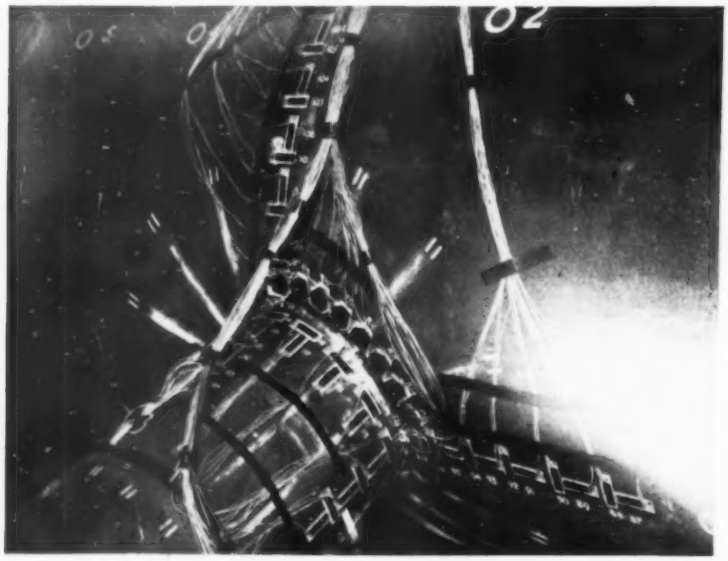
The small mass and the minute size of the electrical resistance strain gauge are two important qualities which make it specially suitable for the measurement of rapidly fluctuating stresses, such as those in vibrating structures or the moving parts of aeroplane engines. The gauges can be attached to the surfaces of moving parts at points which are inaccessible and/or wholly inconvenient for any other form of strain gauge, or they can be scattered extensively over aeroplane structures without giving rise to serious interference with the smooth flow of air over the surfaces.

The measurement of rapidly varying stress necessitates rapid and adequate response in the measuring apparatus, and this can be achieved by using suitable electronic circuits incorporating a cathode ray oscilloscope. The inclusion of a timing circuit of appropriate frequency permits study of the stress-time relationships for stress change frequencies ranging from a few cycles per second to the very high figure of 50,000 or more cycles per second, but for such high frequencies, special precautions against possible errors of measurement must be taken. The possibility of making accurate measurement of stress under high-speed running conditions is of far-reaching importance in engineering design. Machinery failures are usually brought about by dynamic stresses of unknown values which are higher than those permissible under good design conditions. Hitherto, the evaluation of these stresses has not been possible, and the designer has been obliged to rely on methods of trial and error. The electrical resistance strain gauge provides the means of eliminating this unsatisfactory procedure in design.

Method II—The Use of Brittle Lacquers

Contemporary with the development of the electrical resistance strain gauge and its accompanying electronic equipment, there have been important and highly interesting developments in the use of brittle lacquers for the solution of problems of stress analysis and design. The range of usefulness of the method does not altogether cover the same field as the electrical strain gauges, because special attention must be given to the effects of changes of temperature and humidity of the air, and it is usually necessary to work in air-conditioned rooms.

The quality of brittleness possessed by lacquers and resins make them well suited for reacting in a helpful manner under the influence of surface strains produced in stressed parts. The lacquer is carefully sprayed on that part of the surface under examination, and at the same time, a calibration strip of similar material is sprayed and used in a manner described later. The temperature and humidity should be maintained constant if possible during the drying period which occupies about 24 hours. It is also important to point out that the risk of lacquer poisoning must be avoided by making use of gas masks and an efficient ventilating system. After the lacquer has set, the part may be gradually loaded, and when the surface strain reaches some critical value, corresponding to the type of lacquer used, cracks begin to form in directions perpendicular to the greater principal tensile stress, or to the greater principal compressive stress when suitable provision has been made for response to compressive stress. As the strain increases, so the number of cracks increases, and it is possible, therefore, by the aid of the calibration strip, to obtain a fairly good relationship between the stress and the density of cracking over a limited range, but it is the initial cracking which is of chief value.



Courtesy of the British Welding Research Association

Fig. 6.—Resistance strain gauges set up on a branch of a pressure vessel.

For the calibration strip, in the case of structural steel, a thin clean strip of steel, about 10 in. long by $\frac{3}{4}$ in. wide by about $\frac{1}{8}$ in. thick, is suitable; and this can be fixed as a cantilever and a known point load can be applied at the free end as shown in Fig. 7. The uniformly varying strain along the strip produces a corresponding uniformly varying degree of cracking in the brittle lacquer coating. A simple apparatus for carrying out this test is usually supplied with Brittle Lacquer Testing Kits. A simple calculation of the strain or stress at any point along the cantilever makes it possible to correlate stress with the density of cracking, and then, by comparison with the cracking of the lacquer on the part under test, a fair estimate of the stress in the part can be made. If the load is applied to the part in measured increments, the boundary of each zone of cracking can be marked

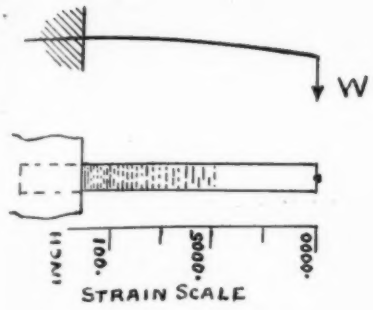


Fig. 7.—Simple calibration strip, showing cracking of the lacquer under the effect of strain.

Fig. 8.—The application of the brittle lacquer technique to a pipe bend. Cracking of the lacquer can be seen in the area enclosed by the white line.

Courtesy of the British Welding Research Association

and labelled with the appropriate stress value. The cracks are not easily seen, but improved visibility can be obtained by applying a preliminary coating of aluminium pigmented undercoat before spraying on the lacquer, and also by applying a red dye etching solution to the lacquered surface after the loading has been applied. With each increment of load, the area of cracking is extended, and new boundary lines corresponding to load increments can be marked. The initial zone of cracking is usually the most important, because, in general, subsequent increment of load only serves to maintain the maximum stresses in this zone. It is often convenient, therefore, to use lacquers of varying stress sensitivity for locating the primary zone of high stress, and for indicating the direction of the principal stresses in this zone. Further interesting and important aspects of this method of stress investigation are briefly referred to below.

Applications of the Method

Casting Design.—Stress conditions in castings are usually complex in form, and the brittle lacquer method makes it possible to locate zones of high stress quickly, and consequently, to accelerate the rate at which design modifications may be made. Prototypes, made to full scale or reduced scale, can be cast in suitable soft metal, and modifications of the casting can be made by removing metal with the aid of portable cutting tools from zones of low stress, or adding metal by welding at zones of high stress.

Dynamic Stresses.—The method has useful applications for studying maximum stress effects of suddenly applied loads—e.g., those brought about by explosives or the impact of masses at high speed.

Compressive Stress.—For the measurement of compressive stress, the load is applied before the aluminium undercoat and the brittle lacquer are sprayed on the surface. After the lacquer has hardened, the load is removed and the relaxation of strain produces cracking of the lacquer at the zones of high compressive stress, and if a lacquer of the appropriate stress sensitivity has been used, a fairly close estimate of the compressive stress can be made.

Critical Strain.—It should be observed that the lacquer begins to crack at some critical strain value irrespective of the stress system in the part. Consequently the maximum stress values will be different in uni-axial and bi-axial stress systems for the same strain indicated by the cracking of the lacquer. It should be remembered, however, that the method has its primary use in locating the zones of high stress, rather than in providing accurate values of the principal stresses.

Stress Measurement in Confined Spaces.—The direct measurement of strain involves reference to a gauge length, and ordinary direct-reading strain gauges take up quite an appreciable amount of room. With the aid of brittle lacquer, however, reliable indications of strain may be obtained in very confined spaces, and successful measurements have been made within a length of 0·010 in.

Residual Stresses.—Manufacturing operations give rise to internal or residual stress of unknown value, and if the part is to be loaded, the additional superimposed stresses may produce stresses which are too high for safe operation. An estimate of the value of the residual stresses may be made by coating the surface of the part with brittle lacquer of known stress sensitivity, and afterwards carefully drilling a small hole of about $\frac{1}{8}$ in. or less in diameter to a depth of about $\frac{1}{8}$ in. The removal of the metal causes a release of strain, and a crack pattern is formed around the hole. Examination of the crack pattern permits a fairly good estimate of the internal stress to be made.

The foregoing applications of the brittle-lacquer method are sufficient to indicate the special advantages of the method in approach to correct design, and at this concluding stage, it is necessary to refer to the special advantages associated with the combined use of brittle lacquer and electrical resistance strain gauges.

Combined Use of Brittle Lacquer and Wire Strain Gauges

Although electrical strain gauges have such important advantages for the analysis of complex stress, there still remains a doubt about the location of the gauges in relation to the zones of highest stress. Location of the

gauges is largely a matter of good guesswork without any means for finding where the zones of high stress lie. It will be observed, therefore, that the brittle lacquer method can be applied for two important purposes as a preliminary procedure—viz :—

1. To locate the zone or zones of high stress.
2. To indicate the directions of the principal stresses in the zones of high stress.

With the aid of this information, the electric resistance strain gauge can be used to the best advantage, and a knowledge of the principal stress directions simplifies stress determination, because two simple straight gauges, set perpendicular to each other can be used instead of the rosette type gauges which must be used when the principal stress directions are not known. It is not always practicable to use brittle lacquer for these preliminary purposes, but where its use is possible, the application of the combined methods provides a powerful aid to closer approach to correct design.

Conclusion

The purpose of this article has been to give a brief description of these two methods and to discuss their bearing on engineering design. Although the methods, in their modern form, were largely developed during the 1939–45 war, the literature relating to them is already large, and in order to assist any readers who may wish to study the subject in more detail, a selected list of references to related literature is given below. The extensive use of electrical resistance strain gauges in the aircraft industry during the war and since, is perhaps the greatest single factor which has contributed to the safety of aircraft in war and peace, and the expanding application of these gauges and the use of brittle lacquers in numerous different fields of engineering manufacture and construction should go a long way to reduce the number of failures that occur in machine and structural parts, and thus diminish the delay and loss of output brought about by the breakdown of manufacturing plant.

In conclusion, the author desires to thank the following :—Messrs. De Havilland Propeller Co., Ltd., Savage and Parsons, Ltd., Baldwins Locomotive International Inc., British Thermostat Co., Ltd. for information relating to strain gauges and equipment, and Messrs. Magnaflux Corporation and the Curtiss-Wright Corporation of U.S.A. for information relating to brittle-lacquer technique.

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Miscellaneous Microchemical Devices (XXI)

An Improvised Micro-Burette

By J. T. Stock and M. A. Fill

BY a little modification, a commercial 1-ml. graduated pipette may be made to function very satisfactorily as a micro-burette. About 40 mm. from the mouthpiece, the stem of the pipette is bent hairpin-wise and then cranked away at right angles, as shown at (*a*) in Fig. 1. After lightly flaring the mouthpiece, a rubber teat *A* is slipped on. The width of the "hairpin" should be such that an ordinary screw clip can be slipped on. Using a small hot flame, a tiny hole *B* is blown near the upper end of the stem. For this purpose, the micro-blowpipe previously developed[1] was found to be very useful. The hole should be squarely situated as shown. By light grinding with fine carborundum paste, the fins of glass raised around the hole during blowing should be removed, so that a small flat area parallel to the face of the "hairpin" is obtained.

A piece of narrow-bore soft rubber tubing *C* is forced on to the swinging bar of a screw clip *D*. The clip is then slipped over the "hairpin" and tightened so that

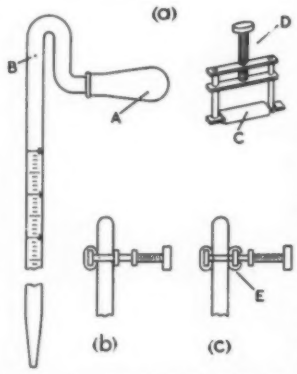

Fig. 1.—Graduated pipette used as micro-burette.

the rubber tubing closes the hole, as shown at (*b*). Liquid may then be drawn into the stem by operating the teat in the usual manner. To deliver, the clip is loosened slightly; in practice, this simple device affords quite a good degree of control.

Another method of controlling is made possible by affixing a strip of soft rubber *E* to the underside of the pressure plate of the clip, as shown at (*c*). The screw is then tightened until the flow of liquid just ceases. Light pressure on the head of the screw then compresses the rubber strip, so that a slight air-leak through *B* allows slow delivery to be made. On releasing the finger pressure on the screw head, the flow stops.

1 Stock, J. T. and Fill, M. A., *Analyst*, 1949, **74**, 122.

Miscellaneous Microchemical Devices (XXII)

Hydrogen Sulphide Supply System

By J. T. Stock and P. Heath
(*Chemistry Dept., L.C.C. Norwood Technical College, London, S.E.*27)

THE generator and distributing system described below were designed to give, with the minimum of attention and wastage, an adequate supply of hydrogen sulphide at a satisfactorily high pressure for semi-micro qualitative analysis.

The generator (Fig. 1) consists of a 500-ml. heavy bolthead flask *A* with a well-fitting, two-holed rubber bung. To prevent blowing out, it is desirable to secure the bung by a loop of soft copper wire that can be easily untwisted for recharging. Through one hole passes syphon tube *B*, the height of which is such that, when the upper end is dipping in diluted (1 : 4) sulphuric acid in beaker *C*, a liquid head of some 50–70 cm. is obtained. The exit tube, to which is attached a gas-washing bottle, a stopcock *D* and a delivery connection *E*, passes through the other hole. Sulphide container *F* is about 8 cm. long, the bore being such that a stick of iron sulphide slides in easily. The upper end of *F* is flared, while the lower end is drawn down to an opening about 2 mm. in diameter.

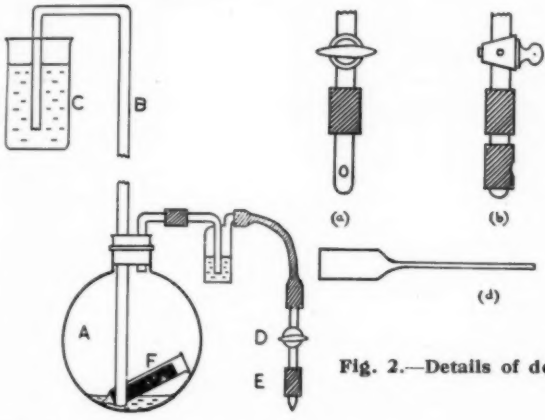

Fig. 1.—Simple hydrogen sulphide generator.

To charge the generator, a stick of iron sulphide some 2 cm. shorter than the container is inserted in the latter. The container is then slipped into the flask so as to take up the inclined position shown in Fig. 1. (Although not usually necessary, the mouth of the container may be supported from the bung by thin platinum wire). By light pump suction at the delivery connection, the syphon is then started, *E* being closed as soon as the acid starts to flow into the flask. Alternatively, beaker *C* may be replaced by a small Büchner flask closed by a bung through which passes the upper end of the syphon tube. The syphon may then be started by blowing at the side-tube of the Büchner flask. After displacement of air, hydrogen sulphide is available as required.

Owing to the inclined position of the container, the wetted area of iron sulphide increases rapidly with the depth of acid in the flask. Accordingly, when operating correctly, this depth is never great. For this reason only a small amount of gas, insufficient to break the syphon action, escapes up the syphon tube. Use of stick-form iron sulphide instead of fragments helps to prevent escape into the acid of small, dissolving particles.

Stopcock *D* is suitably supported on a retort stand or wood block. To the outer end of the tap is attached a short piece of glass tubing, closed at one end and having a hole about 5 mm. in diameter blown in the side, as shown at (*a*) in Fig. 2. A short length of thin-walled, tightly-fitting tubing is then slipped on the tubing, to form a diaphragm over the hole, as shown at (*b*). The point of a sewing needle having been heated to redness, it is thrust centrally through the centre of the diaphragm, so that a tiny perforation is produced. When a sample has to be treated with gas, a piece of fine capillary (about 0·3 mm. outside diameter) is thrust through the perforation, as at (*c*), and after use is withdrawn and thrown away. A stock of capillaries pulled from larger tubing is kept in a covered beaker. This avoids the cleaning necessary with normal, drawn-down delivery tubes (*d*). The likelihood of contamination is thus reduced. Further, a roughly-constant rate of bubbling is obtainable, and, because of the tiny perforation, the accidental opening of the control tap before inserting the delivery tube does not cause an outrush of gas.

An alternative method of forming the diaphragm is shown at (*e*) in Fig. 2. The outer end of the tap, or of a piece of glass tubing connected to it, is heavily flared in the blowpipe. When cold, the glass is wetted and a closely-fitting rubber teat is forced on and drawn tightly as shown. A perforation is then made as before. To assist insertion of the delivery capillary, a small ring of white ink may be described round the perforation, or the latter may be made at the point of intersection of the arms of a white ink cross.

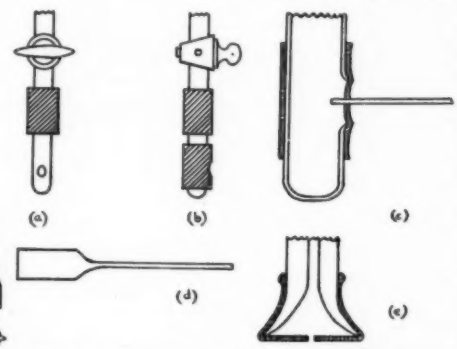

Fig. 2.—Details of delivery connection.

In normal use, the generator operates for several days without requiring recharging. If heavy demand is anticipated, two containers and appropriate sticks of iron sulphide may be placed in the flask. Out of use, the acid head is maintained without apparent leaking for an indefinite period. However, on long standing, some air penetrates into the apparatus, presumably by diffusion through the rubber connections. It is then desirable to purge the apparatus by allowing to run a few minutes.

The perforated-diaphragm type of connection is particularly useful when work by many operators is in progress, thus necessitating multiple delivery points fed from a manifold. The rate of gas-production of the simple generator described above is not normally high enough for feeding several points, and this generator may be replaced by a small Kipp generator adapted for high-pressure delivery. The only modification required is to provide the acid reservoir with a tightly-fitting wired-on bung, through which passes a syphon-feed arrangement similar to *B* and *C* in Fig. 1. The lower end of the syphon tube is situated well down in the stem of the acid reservoir, so that there is no risk of breaking the liquid column of the syphon.

A Method for the Electrolytic Polishing and Etching of Some Al-Ag Alloys, Commercial Purity Aluminium and Pure Magnesium

By L. W. Larke, A.F.R.Ae.S., L.I.M., and Miss E. B. Wicks

Royal Aircraft Establishment, Farnborough

Difficulties having been encountered in the mechanical polishing and etching of aluminium-silver alloys, the authors have developed a solution for the electrolytic polishing and etching of these alloys. Details of the procedure are given and reference is made to the use of the same solution for the polishing of commercial-purity aluminium and pure magnesium.

IT was desired to examine microscopically four binary aluminium silver alloys containing 0·5, 2, 4 and 8% by weight silver, respectively. The alloys were produced from super-purity aluminium and pure silver, a representative analysis being as follows: Ag, 3·99%; Si, 0·008%; Mn, nil; Ni, 0·030%; Cu, 0·004%; Fe, 0·010%; Ti, nil; Al, remainder.

Considerable difficulty was encountered in an attempt to hand-polish the four alloys, in both the "as-received" and heat-treated conditions, by normally recognised practice. Furthermore, the usual etching reagents for aluminium alloys did not satisfactorily differentiate between the solid solution and the precipitated phase, usually producing severe pitting of the surface. Fig. 1 (× 2,000) shows a typical field of the 8% silver alloy solution treated at 450° C. cold-water quenched and aged for 168 hours at 200° C., hand-polished and etched with an aqueous solution of hydrofluoric acid. One of the more interesting difficulties which was encountered was the production of a partially macro-etched surface when using magnesia with either distilled or tap water and blanket cloth. In view of the difficulties and the number of man hours required to produce a rather unsatisfactory hand-polish it was decided to attempt the development of an electrolytic polishing and etching technique.

* All illustrations reduced linear in reproduction.

Apparatus and Method

Electrolytic cells of varying composition similar in some cases to those described in the literature[1, 2, 3] were tried. It was decided to omit from these preliminary experiments electrolytes containing perchloric acid in view of some of their undesirable features. The details of the final and most successful cell to date are as follows:—

The apparatus consists of a 1,000 ml. beaker into which a semi-circular cathode of aluminium is placed. The anode is a piece of No. 12 S.W.G. aluminium wire which carries the specimen (Fig. 2). Electrical contact is established by inserting the aluminium wire into a hole drilled in the back of the specimen. The electrolyte consists of a phosphoric acid-alcohol-water mixture made up as follows:—400 ml. of phosphoric acid of specific gravity 1·75 are diluted with 380 ml. of absolute alcohol to a specific gravity of 1·30, 200 ml. of distilled water are then added, making the final specific gravity 1·25. The potential difference is applied to the cell by means of a simple series resistance circuit. A mechanical

1 Mott, B. W. and Perryman, E. C. W., Electrolytic Polishing of Aluminium Alloys. R.A.E. Report No. M.5838A, Nov., 1943.
2 Waldron, M. B., Middleton, B. F. and Mott, B. W., Electrolytic Polishing of Aluminium Alloys. R.A.E. Report No. Met. 9, June, 1946.
3 Jacquet, P.A., Electrolytic Polishing of Aluminium. *Metallurgist*, April, 116, 1938.

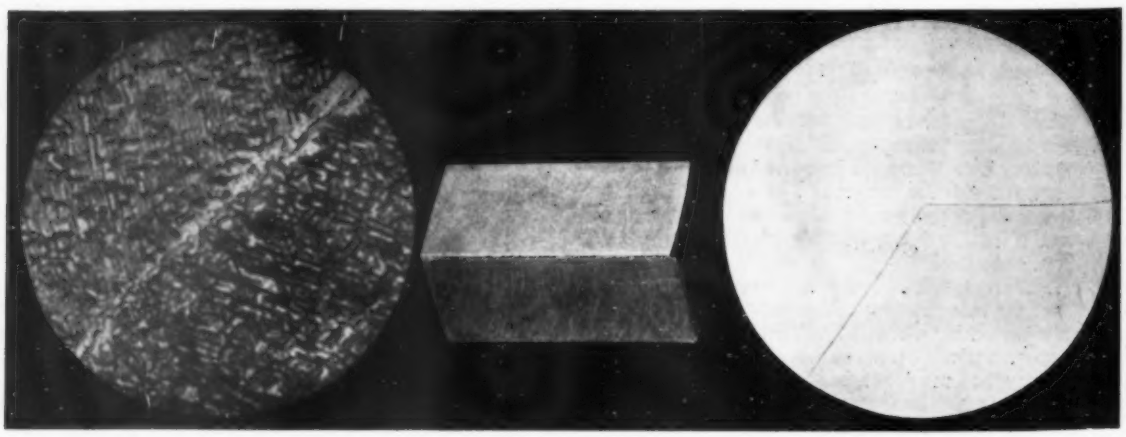

Fig. 1. Fig. 2. Fig. 3.

Fig. 1.—Al-8% Ag alloy, hand-polished and etched in an aqueous solution of hydrofluoric acid. × 2,000.
Fig. 2.—Specimen for electrolytic polishing. × 2.
Fig. 3.—Al-8% Ag alloy solid solution. × 500.

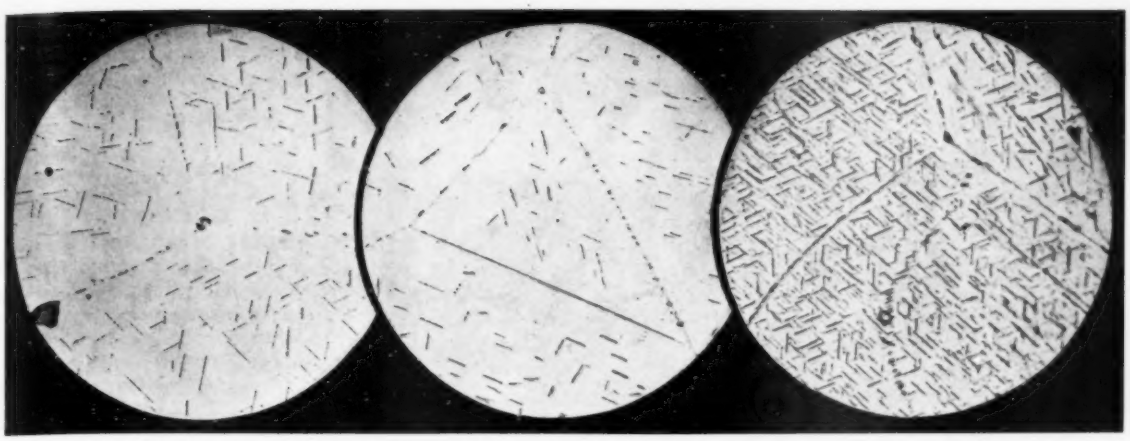

Fig. 4.—Al–2% Ag alloy, aged 250° C. × 500.

Fig. 5.—Al–4% Ag alloy, aged 320° C. × 500.

Fig. 6.—Al–8% Ag alloy, aged 300° C. (compare Fig. 1). × 1,000.

stirrer is situated in the centre of the cell, between the anode and cathode.

The specimens (Fig. 2) which have a total surface area of 10 cm.² are prepared by rubbing down to 000 emery paper followed by a light rub on Velveteen with metal polish. De-greasing is then effected by swabbing in benzol. Immediately prior to immersion in the electrolyte, anode and specimen are rinsed in alcohol to ensure absolute cleanness. The anode is placed in the cell in such a position that the surface of the specimen is $\frac{1}{8}$ in. below and parallel to the surface of the electrolyte. Agitation of the electrolyte is produced by the mechanical stirrer. The precise amount of agitation necessary for any particular cell will vary with the dimensions of the cell and stirrer used and the depth of immersion.

When using a cell with freshly prepared electrolyte a current density of 35 amps./dm.², at a potential difference of 27–30 volts, is found to produce a satisfactory polish (Fig. 3), free from pitting, in from 4–6 minutes when the temperature of the electrolyte is between 42° and 45° C. Current densities of less than 35 amps./dm.² produce an anodic film on the specimens from all compositions; a similar effect is produced when the electrolyte temperature falls below 40° C. Increases in temperature above 45° C. or, alternatively, increases in current density above 35 amps./dm.² both produce prohibitive pitting of the surface. With continued usage, aluminium phosphate is deposited in the cell, the effect of which is to buffer the solution and change its internal resistance, effectively permitting polishing to take place over a greater range of temperatures with the same current density but at a potential difference of the order of 35 volts.

Microscopical examination of a specimen in the solid solution state, polished under the conditions outlined, shows a bright surface with a slight grain boundary etch (Fig. 3). Intensification of the etch may be obtained during the polishing procedure in the following manner. The surface of the specimen is permitted just to emerge from the electrolyte for a few seconds with the current on, the resulting temperature increase of the electrolyte remaining on the surface produces attack. A straw-coloured film may form during this etching treatment but this is readily removed by continued polishing. On completion of the polishing and etching it is important that the specimen be removed from the cell with the current on and immediately transferred to a beaker of hot water to prevent staining. On removal from the hot water the specimen should be rinsed in alcohol and immediately dried in a current of warm air. Figs. 4, 5 and 6 show typical fields of the precipitated phases of the 2, 4 and 8% silver in aluminium alloys after various heat treatments.

Extension of Method to other Materials

The technique as described above has been successfully applied to commercial purity aluminium (99·5% Al) and, with certain modifications, to pure magnesium.

Under similar conditions of current density, polishing times and temperature, specimens of commercial purity

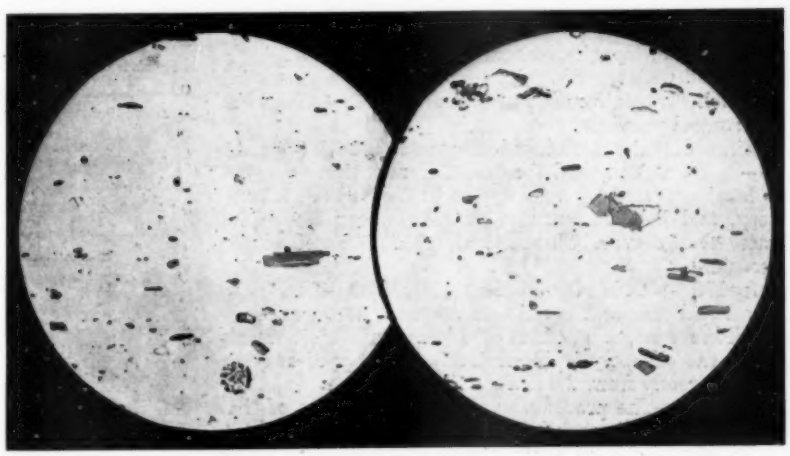

Fig. 7.—Commercial purity aluminium (99.5% Al). × 500.

Fig. 8.—Commercial purity aluminium (99.5% Al). × 500.

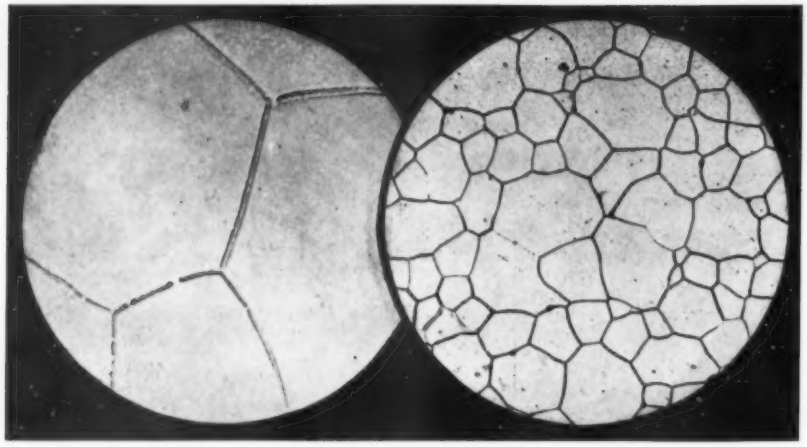

Fig. 9.—Pure magnesium. × 500. Fig. 10.—Pure magnesium. × 100.

aluminium show bright surfaces with impurity constituents etched. The grain boundaries did not become apparent with this material (Figs. 7 and 8).

Specimens of pure magnesium of 10 cm.^{2} surface area were polished and etched in a cell containing the electrolyte previously described (Figs. 9 and 10). In this case the cathode was magnesium and the current density 20 amps./dm.^{2} at a potential difference of 10 volts. The approximate time of polishing was 2 minutes ; no effort was made to control the rise in temperature of the electrolyte. The etch intensification procedure was not necessary with this material. As with the aluminium-silver alloys and commercial purity aluminium, the specimens must be removed from the electrolyte with the current on but, for magnesium, immediate transfer to cold water is essential prior to an alcohol rinse and drying. Should a film form on the magnesium specimens it may be readily removed by swabbing with nitric acid, rinsing in cold water followed by alcohol and finally drying. It may be noted that Jacquet[3, 4] also used a phosphoric acid electrolyte for the electro-polishing of magnesium, but of a different composition from the one described above.

Acknowledgment is made to the Chief Scientist, Ministry of Supply for permission to publish this note. Crown copyright is reserved. Reproduced with the permission of the Controller of H.M. Stationery Office.

4 Jacquet, P. A., The Electrolytic Polishing of Lead-Bronzes, Zinc and Magnesium. *Metaux Corrosion Usure.*, **19**, 71, 1944.

Note on the Estimation of Silver and Copper in Silver Solder

By C. GOLDBERG*

THE determination of silver in silver solder (Ag — Cu — Zn or Ag — Cu — Cd — Zn) is usually carried out by the classical chloride separation. As the chloride ion interferes in the subsequent electrolytic determination of copper, the filtrate is treated with sulphuric acid and taken to sulphur trioxide fumes, diluted, nitric acid added and the resultant solution electrolysed.

The prolonged period of waiting (2 hours—preferably overnight) for complete settling of the Ag Cl precipitate, combined with the necessity for subsequent evaporation to sulphuric acid fumes, makes the process a tedious one for control purposes.

Since Stathis[1] introduced the ascorbic acid (Vitamin C—$C_6 H_8 O_6$) method for the determination of silver, it has been carefully investigated in the author's laboratory, and has been used with satisfactory results for more than a year. The method, which is based on the reaction

$$2 Ag NO_3 + C_6 H_8 O_6 \longrightarrow 2Ag + C_6 H_6 O_6 + 2 HNO_3$$

is a rapid one, and is quite as accurate as the chloride method for larger amounts of silver (above 1%). Moreover, the electrolytic determination of copper may be made directly from the filtrate.

Method.—The procedure used, a modification of that due to Stathis, is as follows :—

Dissolve 1·0g. of silver solder in 10 ml. 1 : 1 nitric acid. If a precipitate of metastannic acid is evident, filter through a No. 42 Whatman (or similar) paper and wash twice with hot water. Evaporate almost to dryness, on a steam bath, and add 20 ml. of distilled water. Heat the solution to about 90° C. and add 60 ml. of the ascorbic acid reagent (2 g. ascorbic acid in 100 ml. of distilled water containing 0·5g. of oxalic acid). Keep the solution warm for 15 minutes and filter through a porcelain filter crucible. Wash with hot water and ignite. Cool and weigh as silver. To the filtrate add 5 ml. sulphuric acid (sp. gr., 1·84) and 3 ml. nitric acid (sp. gr., 1·42) and electrolyse for copper. The ascorbic acid does not affect the quantitative deposition of the copper nor the subsequent determination of cadmium or zinc by conventional methods.

Notes.—Stathis suggested the use of a freshly prepared solution of ascorbic acid because of the ease with which it is oxidised by the air. Since Ponting[2] had found that oxalic acid, in certain concentrations, stabilises ascorbic acid solutions, it was found possible to prepare a reagent which is stable for several weeks. Allowing for a margin of safety, the ascorbic acid—oxalic acid reagent should be freshly prepared every two weeks.

No advantage was found in adding the ascorbic acid solution slowly, as recommended by Stathis.

The silver precipitate invariably settles well within the 15 minute waiting period.

The cost of ascorbic acid (about $1·00 per oz.) is more than offset by the saving in time and the greater convenience of the method.

* New England Smelting Works, Inc., West Springfield, Mass., U.S.A

1 Stathis, E. C., *Analytical Chemistry*, **20**, 271,1948.
2 Ponting, T. D., *Ind. Eng. Chem., Anal. Ed.*, 15, 389–391, 1943.

Primary Analytical Standards

By A. J. Nutten

Department of Chemistry, The University, Birmingham

Continuing the consideration of the various substances which have been advanced as primary analytical standards, the author deals with a further series of substances applicable to acidimetry and alkalimetry.

Potassium Pentaborate

$K_2O.5B_2O_3.8H_2O$. Eq. Wt. 146.63. Weak Acid.
Indicator : Phenolphthalein.

Potassium pentaborate is used for the standardisation of sodium hydroxide[21] though no literature with reference to its use for this purpose has been found.

The substance can be heated to elevated temperatures without loss in water of crystallisation, and it has been found advisable to keep the material at 100° C. for half an hour prior to the preparation of a standard solution.

Atterberg[22] states that potassium pentaborate is stable at 80° C. but that it loses part of its water of crystallisation at 100° C. This loss was not found by others, but it is suggested that the compound be dried at 80° C. for 1 hour.

Standardisation procedure.—After drying and cooling, about 7 g. potassium pentaborate are accurately weighed out and dissolved in warm water in a beaker. When cold, this is transferred to a 250-ml. standard flask and the volume made up to the mark at room temperature ; 50 ml. are pipetted off, about 20 g. mannitol added, and the solution titrated with sodium hydroxide using phenolphthalein as indicator. The potash in the pentaborate is not first neutralised ; consequently there is only one end-point and only four-fifths of the total B_2O_3 is titrated. Potassium pentaborate contains 59·37% B_2O_3 ; hence, the B_2O_3 which is titrated is four-fifths × 59·37%.

Confirmatory tests on the satisfactory results obtained are required before potassium pentaborate is designated a convenient alkalimetric standard of the first order.

Oxalic Acid

$(COOH)_2.2H_2O$. Eq. Wt. 63.03. Weak Acid.
Indicator : Phenolphthalein/Methyl Orange.

Oxalic acid has long been used as a standard in volumetric analysis, but opinions as to its efficacy are sharply divided. Various workers[23] found difficulty in obtaining the compound with exactly 2 molecules of water and uncontaminated by salts. Dodge[1] states that the substance is unsuitable for analytical purposes.

Treadwell and Johner[24] suggested the use of the anhydrous acid which, however, is so hygroscopic that its use as a standard substance is not recommended. The same workers state that the true dihydrate can be obtained by drying the recrystallised and powdered product in a stream of air which has been passed through a mixture of equal parts of hydrated and anhydrous oxalic acid. When so dried, the acid is stated to yield results comparable to those obtained with other standards.

It is of interest to note that it is possible to titrate oxalic acid to methyl orange or methyl yellow if an excess of calcium chloride is present.[25] Calcium oxalate is precipitated, and the hydrochloric acid also formed is titrated with alkali :—

$$CaC_2l + H_2C_2O_4 \longrightarrow CaC_2O_4 + 2HCl$$

Though oxalic acid may be used for ordinary analytical purposes, its suitability in very accurate work must be questioned. A low equivalent weight increases the error of weighing.

Potassium Chlorate

$KClO_3$. Eq. Wt. 20.43 ——→ Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

The use of potassium chlorate as a standardising substance for solutions of alkali was suggested by van Valkenburgh,[26] who claimed uniform results agreeing closely with those obtained by standardising against standard acid. The pure chlorate is easily obtained by recrystallisation of the ordinary C.P. salt followed by drying for 4–6 hours at 240° C.

For standardisation purposes the solution of potassium chlorate is boiled in an open Erlenmeyer flask and sulphur dioxide passed into the boiling solution for 30 minutes. The excess sulphur dioxide is boiled off (5–10 minutes) and the solution titrated against the alkali using phenolphthalein as indicator.

$$3SO_2 + KClO_3 + 3H_2O \longrightarrow 3H_2SO_4 + KCl$$

Though the results obtained by van Valkenburgh have not been questioned, the method is tedious and not practicable for ordinary analytical work.

Potassium Bitartrate

$KHC_4H_4O_6$. Eq. Wt. 188.18. Weak Acid/——→Weak Base.
Indicator : Phenolphthalein.

Potassium bitartrate[27] is sparingly soluble in cold water, hence titration must be carried out in hot solution with phenolphthalein or thymol blue as indicator.

The substance may be used for titrating acids, being converted to potassium carbonate prior to its use, but is not recommended as a primary standard due to the difficulty encountered in testing its purity accurately.

Adipic Acid

$(CH_2)_4(COOH)_2$. Eq. Wt. 73.07. Weak Acid.
Indicator : Phenolphthalein.

Van Voorst[28] claims that pure adipic acid for use in alkalimetry can easily be prepared by recrystallising the commercial product from five times its w ight of distilled water, and drying at 130° C. Meyling[29] confirms this work and mentions that adipic acid is fairly cheap.

Adipic acid is not very soluble in water (1·4 g./100 g.) but is used extensively in alkalimetry.

Salicylic Acid

$C_6H_4(OH)(COOH)$. Eq. Wt. 138.12. Weak Acid.
Indicator : Phenolphthalein.

Rosicky and Tamenchyma[30] state that salicylic acid is a satisfactory primary standard. The solution in 50%

ethanol can be titrated with sodium hydroxide using methyl red or phenolphthalein as indicator. The sparing solubility of the compound is a disadvantage.

Skramovský[31] also claimed salicylic acid to be a standard substance, but his results do not substantiate his claim.

o-Chlorobenzoic Acid

$C_6H_4Cl(COOH)$. Eq. Wt. 156.57. Weak Acid.
Indicator : Methyl Red.

Murgulescu and Alexa[32] describe a method for titrating *o*-chlorobenzoic acid with alkali, and claim constant results. They prefer this acid to salicylic acid.

p-Nitrobenzoic Acid

$C_6H_4NO_2(COOH)$. Eq. Wt. 167.12. Weak Acid.
Indicator : Phenolphthalein.

Thornton and Getz[33] investigated the properties of the acid. It is readily purified, two or three recrystallisations from ethanol being generally required. It is not bulky and is of high molecular weight ; it requires drying at only 120° C. and is not excessively hygroscopic.

The acid has not come into common use in neutralisation processes and would appear to be unsuitable for accurate work.

Maleic and Fumaric Acids

$(CH.COOH)_2$. Eq. Wt. 58.04. Weak Acids.
Indicator : Phenolphthalein.

A paper by Lange and Kline[34] describes the use of these acids as alkalimetric standards. Good results are claimed. The acids cannot be used to titrate solutions of ammonia.

Succinic Acid

$(CH_2.COOH)_2$. Eq. Wt. 59.04 Weak Acid.
Indicator : Phenolphthalein.

Ljungren[35] claims that correct results may be obtained by using succinic acid to standardise 0·1N alkalis, provided the acid is dissolved in a very little carbon dioxide-free water.

Kolthoff[36] does not recommend succinic acid as a primary standard due to the ease with which it is converted to the anhydride on drying.

Malonic Acid

$CH_2(COOH)_2$. Eq. Wt. 52.03. Weak Acid.
Indicator : Phenolphthalein.

Malonic acid has been proposed as a standard substance by Phelps and Weed,[37] but it is difficult to prepare in an analytically pure state.

Malic Acid

$COOH.CH_2.CH(OH)COOH$. Eq. Wt. 67.04. Weak Acid.
Indicator : Phenolphthalein.

Lange and Kline[34] claimed that malic acid gave results of high accuracy when used as a standard for sodium hydroxide solutions. It cannot be used as a titrant for solutions of ammonia.

4-Amino-2-Sulphobenzoic Acid

$C_6H_3NH_2(SO_3H)(COOH)$. Eq. Wt. 108.6. Weak Acid.
Indicator : Phenolphthalein.

2-Nitro-4-Sulphobenzoic Acid

$C_6H_3NO_2(SO_3H)(COOH)$. Eq. Wt. 123.59. Weak Acid.
Indicator : Phenolphthalein.

4-Nitrotoluene-2-Sulphonic Acid

$C_6H_3CH_3(NO_2)(SO_3H)$. Eq. Wt. 217.19. Weak Acid.
Indicator : Phenolphthalein.

These three acids were proposed as standards by Kastle.[39] All are claimed to be preparable in a pure state and to give a sharp end-point with phenolphthalein.

Pseudo-Cumene Sulphonic Acid

$C_9H_{11}SO_3H.2H_2O$. Eq. Wt. 236.28. Strong Acid
Indicator : Phenolphthalein/Methyl Red.

This acid has been claimed by Tischenko[38] to have a very small vapour pressure despite its hydrated state, and to be suitable for the standardisation of alkalis. The substance has not been widely used as a standard.

Picric Acid

$C_6H_2OH(NO_2)_3$. Eq. Wt. 229.11. Weak Acid.
Indicator : Ethyl bis- (2 : 4-dinitrophenyl) acetate.

Picric acid, recommended as a standard by Sander,[40] is likely to contain organic impurities, and its use as an alkalimetric standard is not desirable. The above indicator, available commercially, was proposed by Fehnel and Amstutz[41] for the titration of orange- and red-coloured solutions in which the end-point with phenolphthalein is difficult to detect.

Ascorbic Acid

$C_6H_8O_6$. Eq. Wt. 176.12. Weak Acid.
Indicator : Phenolphthalein.

Rosenthaler[42] reports that this compound gives satisfactory results when used as an alkalimetric standard. As yet this work has not been confirmed, but it is certain that the compound will not come into general use as a standard due, among other things, to its expense and to the fact that there are many more readily obtainable standards.

Hydrazine Sulphate

$(NH_2)_2.H_2SO_4$. Eq. Wt. 65.06. Strong Acid.
Indicator : Methyl Red.

Kolthoff[43] states that this salt may be readily purified by recrystallisation from water, is anhydrous, and is strongly acid. Good results have been obtained using methyl red as indicator.

Aniline Sulphate

$2C_6H_7N.H_2SO_4$. Eq. Wt. 142.162 Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

o-Chloroaniline Sulphate

$2C_6H_6ClN.H_2SO_4$. Eq. Wt. 176.61. Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

p-Anisidine Sulphate

$2C_7H_9ON.H_2SO_4$. Eq. Wt. 172.19. Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

p-Toluidine Sulphate

$2C_7H_9N.H_2SO_4$. Eq. Wt. 156.19. Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

Strasser[44] has studied the above salts and has proposed them as standard substances. His results have not been confirmed and the compounds have not yet been established in volumetric analysis.

Pyridine Perchlorate

$C_5H_5N.HClO_4$. Eq. Wt. 179.56. Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

Arndt and Nachtwey[45] suggested the use of this salt as a standard, but confirmation of their results is not available.

Potassium Tetroxalate

$KHC_2O_4.H_2C_2O_4.2H_2O$. Eq. Wt. 84.73. Weak Acid.
Indicator : Phenolphthalein.

This salt has been recommended by several workers as an alkalimetric standard,[46] but others[47] state that it is not suitable. In view of the conflicting results obtained it is not surprising that the substance is not widely used in alkalimetry.

Potassium Acid Oxalate

KHC_2O_4. Eq. Wt. 128.12. Weak Acid.
Indicator : Phenolphthalein.

Osaka and Ando[48] state that potassium acid oxalate is suitable for standardising solutions of alkali. Unfortunately, the salt is not easily prepared in a pure state ; for example, it cannot be recrystallised directly due to the formation of the tetroxalate.

Cadmium Sulphate

$CdSO_4.8/3\ H_2O$. Eq. Wt. 128.26. ——→ Strong Acid.
Indicator : Phenolphthalein/Methyl Red.

This substance, when electrolysed in solution, yields an equivalent quantity of sulphuric acid[49] according to the equation :—

$$CdSO_4 + H_2O \longrightarrow Cd + \tfrac{1}{2}O_2 + H_2SO_4$$

Kolthoff states that the procedure is of interest only from the point of view that it gives a method for the preparation of standard sulphuric acid, and is unlikely to replace the more convenient methods for alkali standardisation.

Cyanogen Bromide

CNBr.

This compound has been used by Møller[51] for the standardisation of acids and alkalis. It is claimed to be readily obtainable and preserved in a state of analytical purity. Its use as a standard depends upon the reactions :—

$$BrCN + 2I^- + H^+ \longrightarrow Br^- + HCN + I_2$$

$$BrCN + 2OH^- \longrightarrow Br^- + OCN^- + H_2O$$

The unpleasant odour of the compound and its tear-producing effect make it inconvenient to use, and it is not recommended as a standard.

Silver Nitrate

$AgNO_3$.

At a *p*H of 4·2 a mixture of mercuric cyanide and silver nitrate gives a clear solution.[52] The addition of alkali precipitates silver cyanide and, when all the silver nitrate is consumed, the next drop of alkali may be detected with phenolphthalein indicator. This reaction has been made the basis of a procedure for standardising solutions of alkali, but further investigation is necessary before the method is accepted.

Sodium Carbonate

Na_2CO_3. Eq. Wt. 53.00. Weak Base.
Indicator : Methyl Orange.

The sodium carbonate, obtained by heating C.P. sodium bicarbonate to 375° C. followed by washing with ethanol and drying at 110° C. is claimed by Waldbauer, McCann and Tuleen[53] to be satisfactory as a volumetric standard.

The experiments of Smith and Croad,[54] however, indicate that it is inadvisable to heat the bicarbonate above 300° C. as sodium carbonate decomposes appreciably at 310°–315° C. These workers state that sodium carbonate prepared from pure sodium bicarbonate with vacuum dissociation at 300° C. is suitable for use in acidimetry.

Sodium carbonate is slightly hygroscopic, and even takes up moisture during weighing. Kolthoff[55] noted an increase of 1–3 mg. while weighing 2·65 g. in a weighing bottle, and, as a result, considerable errors may result on titration.

Storage of sodium carbonate is recommended in glass-stoppered bottles in a desiccator over anhydrous magnesium perchlorate. The substance is not recommended for the most accurate work.

Reference may be made at this stage to a paper by Calcagni and Sturnajolo,[56] who tried various substances as standards for acids. They state that potassium, lithium, calcium and barium carbonates, in addition to calcium and magnesium oxides, and sodium, potassium and magnesium metals are all simpler to use than sodium carbonate and sufficiently precise.

Borax

$Na_2B_4O_7.10H_2O$. Eq. Wt. 190.72. Weak Base.
Indicator : Methyl Orange/Methyl Red.

The one disadvantage which has prevented the extensive use of borax as a standard substance is the difficulty encountered in obtaining the dry salt.

Air-drying has been recommended by Lindfors[57] but this is not satisfactory, because the solution remaining on the surface of the crystals is sufficiently alkaline to absorb carbon dioxide from the air with formation of sodium carbonate or bicarbonate as impurities.

Kolthoff[58] recommended drying over moist sodium bromide, but this salt is relatively expensive and the drying process is slow, rendering the method, to a certain extent, unsatisfactory. The difficulty was overcome by Hurley[59] who recrystallised borax from water at a temperature below 55° C. to prevent formation of the pentahydrate, filtered the crystals by suction, and washed with water. The borax crystals were dried by washing with ethanol then with ether. On evaporation of the ether the pure decahydrate was obtained.

Perhaps the best drying procedure is that of Menzel[60], who spread the damp crystals on a watch-glass and placed it in a desiccator over a solution saturated with respect to both sucrose and sodium chloride. Hurley carried out tests with two borax samples prepared as above and dried by his alcohol-ether method. His results were comparable with those obtained using a sample dried by Menzel's method and with a pure sodium carbonate sample.

There is no doubt that borax prepared and dried as above is suitable for use in acidimetry.

Potassium Bicarbonate

$KHCO_3$. Eq. Wt. 100.11. Weak Base.
Indicator : Bromocresol Green.

Van Than[61] recommended potassium bicarbonate as a primary standard substance. A product with 100% activity has been prepared by Incze[62] who passed carbon dioxide into an ethanolic solution of potassium hydroxide, dissolved the precipitate in water and reprecipitated with alcohol.

The substance is anhydrous and non-hygroscopic, and is strongly recommended as a standard substance by Incze. Kolthoff and Sandell[63], using the method of Schmitt[64] for the preparation of the compound, dried the salt over concentrated sulphuric acid in an atmosphere of carbon dioxide. They found that the product gave a titre agreeing with that obtained with borax, sodium carbonate, and a sample of potassium

Continued on page 180

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The Sunvic D.C. Amplifier (Type D.C.A.1.) has been developed to fulfil these functions. As it embodies no moving parts, beyond a vibrator, it is quite robust and unaffected by vibration. The speed of measurement or recording is determined mainly by the characteristics of the final instrument used in conjunction with it and the four-position range-switch provides rapid and easy means of selecting the input level.

By providing a source of standard voltage to back off the e.m.f. of the measuring element, and feeding the out-of-balance voltage into the amplifier, it is possible to record the deviation from a desired value. For some applications, such as the measurement of furnace temperature in creep-test work, this is particularly advantageous as it enables a full-scale record to be obtained for a temperature change which is extremely small in relation to the operative temperature.

The stability and speed of the amplifier makes it convenient to use in conjunction with auxiliary switch-gear to measure or record the output of a number of thermocouples or strain gauges as opposed to the more tedious method of measurement by potentiometer. It can also be used as a reference voltage source which is continuously standardised against a standard cell to one part in 10,000.

The D.C. input is connected to a vibrator which changes the D.C. voltage into an alternating square wave. After suitable A.C. amplification, the amplified signal is rectified and connected either to a D.C. milli-ammeter or to a recorder. A high degree of negative feed-back is applied, giving high stability and linearity. The degree of feed-back is such that the overall gain is determined entirely by the feed-back resistance.

After a stable operating temperature has been reached in the amplifier, the long-term stability over 1,000 hours is ± 1 micro-volt where the mains voltage variations do not exceed ± 6%. Short-term stability over periods of about 5 hours is ±0·2 micro-volts. Mains voltage variations of ± 12% introduce an error of approximately ± 2 micro-volts.

The instrument is provided with four ranges giving full-scale deflection on a 5-0-5 milliammeter for 100 millivolts, 10 millivolts, 1 millivolt and 100 microvolts, and the maximum speed of response is 0·1 sec. full scale. A high-speed recorder can be supplied having a full-scale deflection for ± 5 milliamps, the maximum chart speed being 12 in./hr. and writing speed 35 cm./sec.

Where it is proposed to use the amplifier to measure the deviation of an e.m.f. from a desired value, a source of standard voltage is necessary. For such use the Sunvic Standardising unit (Type M.S.U.1.) has been developed. This is essentially a source of continuously variable voltage, of low e.m.f., which is manually standardised against a built-in standard cell. A ten position switch allows selection of voltage in approximately 5 millivolt steps, with a potentiometer which more than covers each of these steps.

Sunvic Controls, Ltd., 10, *Essex Street, Strand, London, W.C.2.*

Book Reviews

INDENTATION HARDNESS TESTING

By Vincent E. Lysaght, pp.288, 135 illustrations. 1949. New York $5.50. (London Chapman & Hall, 44/–)

THE non-destructive testing of materials is a subject of great interest to producers and scientists at the present time. The static indentation hardness test occupies a prominent position in this field of examination, and has become of great importance. The quality control of products such as hard steel safety razor blades of only two thousandths of an inch thickness presents many problems, and yet it is now possible to obtain quantitative results on articles of this kind. Furthermore, with the introduction of hardness microtests, we can control the quality of parts for watches, and this book describes hardness indentations made on pivots which are only ten thousandths of an inch long and five thousandths of an inch in diameter. During the first war, Brinell ball testing became very important and, as a French writer once put it, was covered with military glory. The last war also earned medals for indentation testing, and we are told in the book under review that 50,000,000 hardness tests a day were made in the United States.

A warm welcome can be given to a work produced by an author who has been for 20 years on the staff of the Wilson Mechanical Instrument Co., which makes the various Rockwell machines. The title of his book is rather general, but in fact it deals only with static tests, largely because E. Meyer suggested that dynamic effects should be eliminated from the notion of hardness. Nine pages, however, (starting on page 48, and not page 46 as given in the index) are devoted to the Scleroscope, but the use of this particular instrument for the difficult problem of the testing of the hardness of rolls is not emphasised, although a certain amount of the text deals with portable testers and commercial apparatus intended to deal with special requirements. This part of the work is not very critical and does not provide us with much evidence as to the actual value of results obtained from various types of apparatus. On page 132, for instance, we learn of some standard steel scratch pins marketed by an English firm, but we do not learn whether the use of these pins is actually a reliable method of hardness testing. The reviewer's experience suggests that it is a very difficult procedure where very much depends upon the sensitivity of the operator. On this page there is also mention of the Poldi test, but there must be a slip in the text and "diagonals" of the impressions should read "diameters."

Regarding scratching tests, we are given a few details about the file test, and the following values on the Rockwell scale are submitted: Special Testing Files C67; limit of fileability C64; untempered high-speed steels at C62 are not fileable (which probably indicates that they contain some austenite of high work-hardening capacity). These files will cut high-carbon steel quenched and tempered to C63, but will not appreciably scratch steels of C65, whilst it is stated that they bite slightly on material of C63–C61. In this work we are entering the realm of what Scott Blair has called psychotesting effects, which play a part in the study of materials like cheese and dough. Whilst considering scratching, it may be mentioned that various glasses have indentation hardness values ranging from 373–546 Kg./sq. mm.

(Knoop test), and the book contains important information on the testing of plastics, non-metallic materials, minerals and abrasives.

A section of the work of particular value is the 50 pages or more devoted to the development and behaviour of the various Rockwell instruments. The Vicker's diamond pyramid test naturally does not receive the same amount of attention, but satisfactory descriptions are given of the ball test in its various forms, together with a chapter on how to carry out Meyer hardness analysis. There is reference to the latter as related to the Rockwell test, and a special feature is the provision of up-to-date information on approximate conversions of hardness scales. We are also supplied with an alphabetical list of common engineering tools giving the composition of steels from which they should be hardened. One will gather from this survey that the book chiefly aims at giving technical information on the problems with which the engineer and the metallurgist engaged on hardness testing must cope. No attempt has been made to consider hardness from the theoretical standpoint, and so only a few references to the literature are given. The work includes very extensive tables for the determination of hardness values on various scales, and this makes it a compact but comprehensive handbook which can be recommended to engineers and metallurgists.

MULTIPLE-BEAM INTERFEROMETRY OF SURFACES AND FILMS

By S. Tolansky; 187 pp., 113 diagrams and illustrations. Published by Geoffrey Cumberlege, Oxford, at the Clarendon Press, July, 1948. English price, 18s.

THIS book, the first volume of a series entitled "Monographs on the Physics and Chemistry of Materials," describes the new optical interference techniques developed by the author since 1942. They will undoubtedly find application in many fields including metal physics, and since some metallurgists may be unfamiliar with Professor Tolansky's work, a brief description is not unwarranted here. The techniques described are delicate refinements of ordinary interference methods; but it is shown that by using multiple reflections the fringes are made very sharp so that surface features only a few Angstroms in height are seen and measured. The magnification in depth up to $\times$ 500,000, is comparable with the magnification in area given by the electron microscope.

Multiple-beam Fizeau fringes and their application are discussed (Chapters IV to VII). The numerous photographs leave no doubt as to the power of the method and its superiority over previous interference techniques. Crystal surfaces are shown crossed by fine fringes, effectively contour lines, which indicate clearly the most minute surface irregularities. Chapters VIII and IX deal with a novel multiple-beam system producing "fringes of equal chromatic order." These give a direct picture of extreme sensitivity of the profile of the surface.

Chapter XII on opaque substances and metals will directly interest metallurgists. The study of metallic surfaces and the measurement of thin metal films are discussed. Chapter XIII considers the production of accurate plastic replicas with the examination of metal surfaces particularly in mind. Most of the last two chapters, however, concern purely optical matters.

A simple but adequate account of the optical theory is given together with details of apparatus and practice. One pleasing feature is the simplicity of the apparatus used, in view of the remarkable results obtained. It must be emphasised that this is not a book in which metallurgists will find a store of new information about metals.* Rather is it an introduction to a striking new technique which the research worker should find of considerable value in his own laboratory. It may, therefore, be recommended; for as well as providing details of experiment it is careful to advise on the natural limitations of the technique and on pitfalls the beginner may meet. In addition, this book will be a necessity for those who wish to follow and appreciate the very recent work of Tolansky's school on hardness tests, abrasion tests and slip-bands.

Altogether this is a most readable volume, the subject being presented with clarity and charm. The excellence of the illustrations, and the printing and production combine to make the price of 18s. most reasonable.

J. R. B.

* It is not, of course, devoted exclusively to the study of metals.

Primary Analytical Standards

Continued from page 177

bicarbonate prepared according to Incze, to within 0·02%.

Potassium bicarbonate is, then, well-suited for the standardisation of acids.

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